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INFLUENCE OF THE RESERVOIR MATRIX ON THE MECHANISM AND KINETICS OF THE IN SITU COMBUSTION PROCESS FOR HEAVY OIL RECOVERY

VOLUME I OF II

by

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1. OBJECTIVE

The overall objective of this proposal is to obtain a better definition of the mechanism of heavy oil recovery by the in situ combustion process, through improved measurement of fuel laydown kinetics.

Specific objectives of this proposal are:

(i) Quantitative determination of the effects of the reservoir matrix on the kinetics of fuel laydown.

(ii) Definition of the extent to which coke devolatilization reduces the fuel available for combustion.

(iii) Correlation of the kinetics obtained by TGA methods on fuel laydown and coke devolatilization with those obtained above.

(iv) Provision of improved kinetic parameters as input to numerical simulations of the in situ combustion process.

2. JUSTIFICATION

The In Situ Combustion process is now a frequently used method for the production of heavy oils, and research on its application to tar sands is currently in progress. The process is thermally more efficient than use of steam injection and can give high oil recoveries. The number of pilots and commercial operations will increase as production from light oil reservoirs declines. Development of a better understanding of the mechanism of the process is needed.

Pilot design is usually based on combustion tube results (air and fuel requirements, fire front advance rates, etc.) coupled with subsequent use of a numerical simulation tuned by the combustion tube run.

The quality of results from kinetically based simulations is dependent on the quality of the input data. In the case of the kinetic data this is currently very poor since much of the data was not derived with this process in mind. Indeed developments in mathematical computational techniques have far outpaced knowledge of the mechanism and the basic chemical data on which the technique is based. In particular, coke laydown kinetics used in simulations have very different temperature dependencies from those measured experimentally, and coke devolatilization has been ignored entirely in consideration of the process mechanism.

Improved definition of the process details and better quantitation of the kinetic variables involved will provide more accurate simulation results. These will result in more confident assessment of the application of the process to a given reservoir, improved oil recoveries, and decreased cost of oil production.

3. SUMMARY

The objectives of this work have been to determine if the remarkably low activation energies for coke formation that are employed in numerical simulations of firefloods are due to clay catalysis, and to evaluate the significance of coke devolatilization on fuel availability.

Experimentally, the effect of clay on coke formation in in situ combustion has been studied by two techniques:

1. by the use of isothermal reactors operated at pressures and temperatures corresponding to those in a reservoir in the region of the firefront, with either Pelican or Delmas crude oils and

2. by non-isothermal thermogravimetric analysis of smaller samples at atmospheric pressure employing Pelican vacuum resid in place of total crude.

In both parts of this work, the amount of coke formed has been measured, in the isothermal reactor case by sample combustion and measurement of carbon dioxide formed, and in the TGA work by mass loss on combustion.

The isothermal reactor studies employed sample sizes of approximately 200 g, of which 20 g was Pelican crude and the remaining 180 g was either Ottawa sand or sand containing a clay. The clay present was either kaolin, montmorillonite, or illite and, when present, constituted 10% by mass of the total.

The reactors employed were constructed from stainless steel, were operated at 500 psi and temperatures in the range of 380-435°C by submersion in a sand bath. A flow of nitrogen gas through the

reactor during the experiments simulated the gas flow through the coke-formation region of the reservoir downstream of the combustion front in a fireflood. Duration of these experiments ranged between 2 and 8 hours. The amount of coke formed in each experiment was determined from a composite sample of the synthetic core taken after each run. To determine the kinetics of coke formation for each clay type runs were performed at three reaction times and four reaction temperatures. Graphs of the natural log of the rate constant versus the reciprocal of the absolute temperatures were employed to provide activation energies for coke formation.

The TGA work studied the effect of the three clays above, at levels ranging from 10-90%, on both the amount of coke formed and the kinetics of thermal decomposition of Pelican Resid. This work was also performed in the absence of oxygen, but in contrast to the isothermal reactor work was at atmospheric pressure. Sample sizes employed were approximately 40 mg. For each sample type employed, non-isothermal TGA runs were performed at four heating rates ranging from 5 to 20°C/min. Coke formed in the course of each run was determined by switching the purge gas from nitrogen to air at 650°C and measuring the mass loss ensuing. Activation energies for decomposition of the resid were determined using either the DuPont software accompanying the TGA equipment or by a method due to Friedman.

Coke devolatilization was examined by TGA using coke samples taken from the larger scale work and measuring the loss occurring when the samples were heated between 400 and 600°C. An estimate of the kinetic activation energy for the minor losses observed was also made using single heating rate data.

NOTE: For convenience this work is reported in two volumes. Volume 1 presents all the experimental work, discussion and conclusions, and key tables and figures. Volume 2 is primarily a set of appendices in which, for completeness, the TGA raw data is reported.

4. CONCLUSIONS AND RECOMMENDATIONS

1. At clay levels representative of those encountered in a reservoir (≈10%) large reductions in activation energies for either coke formation or crude thermal decomposition are not observed.

2. Clays are not responsible for the discrepancies between activation energies in fireflood numerical simulations and those found by experimental measurement.

3. The relationship between block size in numerical simulations, degrees of history matching and activation energies for coke formation should be examined.

4. Thermogravimetric analysis (TGA) methods at atmospheric pressure do not accurately simulate larger scale studies performed isothermally and, more importantly, at higher pressures.

5. It is known that coke formation is influenced by pressure, therefore TGA work at high pressure should be undertaken on simulated core samples to determine if better agreement with the large scale high pressure experiments can be achieved.

6. The isothermal reactor work showed that activation energies for coke formation are not discernibly reduced by clays present at the 10% level, relative to the presence of Ottawa sand alone. This is in general accord with recent literature results.

7. The TGA work demonstrates that a single value of activation energy is insufficient to describe the thermal decomposition of a heavy oil resid; activation energies increase with percent decomposition and reach a maximum value.

8. Thermal decomposition activation energies measured by TGA are reduced by the presence of clays; however, large (50%) amounts of clay are required to produce meaningful effects.

9. Isothermal reactor studies found that illite was clearly superior to either kaolin or montmorillonite in promoting coke formation. The TGA technique in contrast did not distinguish between the three types of clays.

10. Under isothermal reactor conditions, clays promoted coke formation by increasing rates of coke formation and not by altering activation energies.

11. The amount of coke formed in thermogravimetric work is clearly dependent on heating rate. This difference is attributed to rapid loss of volatiles at high heating rates which would otherwise contribute to coke formation.

12. Comparison of results from Pelican and Delmas crudes shows that under isothermal reactor conditions, Delmas crude produces more coke in agreement with its lower API gravity and higher Ramsbottom carbon content.

13. Both Pelican and Delmas crude in the presence of illite clay produce substantially less coke than reported for Athabasca bitumen with or without sand/clay.

14. Devolatilization of coke is not a significant occurrence. TGA studies on coke from isothermal reactor work shows that mass loss does not generally exceed 10% between 400 and 600°C.

5. LITERATURE REVIEW

5.1 Coke Formation - General Features

The purpose of this section is to review the literature to date on fuel formation in in situ combustion, and to provide a basis for the work undertaken in this contract. In practice, fuel requirements for design of a fireflood pilot are obtained experimentally from combustion tube work. The equipment and ancillary facilities for this type of work are extremely expensive and relatively few companies can afford the cost of maintaining and operating them. Numerical simulation of the same process represents a potentially cheaper alternative or can be used in conjunction with combustion tube results to develop a better insight into the progress of an ongoing fireflood, or modify the design of one in the planning stages. Many of these simulators are kinetically based, i.e. they require input data on the rates of fuel formation, where fuel is generally regarded as the coke formed by pyrolysis of the crude.

In this section we try to show that there is very little and perhaps no data on coking kinetics that can be used with much confidence as input to numerical simulations. There are two main reasons:

1. The vast majority of the numbers commonly employed and/or cited have not been generated for the purpose of modelling in situ combustion. Thus simulation users have had to use what has been available and the applicability has been questionable.

2. The discretization of a combustion tube or reservoir into blocks for simulation may well be enforcing the use of incorrect numbers to enable the simulator to run. The kinetically significant region is probably only a few inches in length whereas blocks are seldom less than six inches long.

These two points will be elaborated on later in this section but an example at this time will provide a useful illustration. Bennion and Hayashitani (1) undertook an extensive study of the kinetics of coke, light and heavy oil and asphaltene concentrations in the thermal cracking of Athabasca bitumen. Their experimental study necessarily differed somewhat from true reservoir conditions - the system was a closed one (autoclave), there was no reservoir sand present and no gas flow, but it was an attempt to obtain results specifically for simulation work. For coke formation, the activation energy was found to be 44-47 kcal/mole, indicating that coke formation was significantly temperature dependent. Bennion subsequently (2) attempted to use these results to history match a combustion tube run with the ISCOM model. These attempts were quite unsuccessful (coke/fuel laydown was insufficient) and his autoclave work was essentially discarded in his modelling. Asphaltenes were still considered as the source of the coke but the activation energy was reduced to 17 kcal/mole, i.e., a very low temperature dependence and their concentration was varied to give the observed fuel concentration. Thus, either the simulator could not handle 'good' kinetic data or Bennion's original results were incorrect. In this particular case the truth probably lies in between.

5.2 Coking Kinetic Data

In Table 1 is collected virtually all of the kinetic data currently reported as being used in simulations, together with some recent results (those of Schucker and Barbour) which have not yet been so employed. Comments on the reasons for the work, appropriateness, and methods are given where considered useful. It is worth noting that the numbers employed by Crookston, who developed one of the first models for in situ combustion are taken from the literature on low temperature oxidation. Thus Crookston is either implicitly assuming that all fuel arises from LTO (low temperature oxidation) ahead of the combustion front or used these values because none other were available. Thomas is the only author who explicitly makes this (LTO) the basis for his work. The present consensus of opinion, judging from published work, is that fuel arises from cracking reactions, combined with gas and steam stripping in the region in front of the combustion zone. Consequently, along these lines it is common to divide the zones of a fireflood into (a) a burned zone, (b) combustion zone, (c) cracking zone, (d) evaporation and visbreaking zone (e) steam plateau, (f) water bank, (g) oil bank and (h) initial zone. It has not been established that the fuel burned is solely coke as defined as toluene-insoluble carbon. A better definition may be that the fuel is the material (coke/heavy ends) which remains at the approach of the firefront, and is consumed.

Activation energies in the Table fall into two groups:

(i) 35-50 kcal/mole: most of these are the experimentally determined set, but with the exception of Bennion's work, none involve actual measurement of 'coke', most are true measurements of bitumen cracking. Few published simulations employ such high activation energies. Culham has done so, terming them 'non-catalytic' but his pre-exponential factors look as if they have been increased to achieve sufficient fuel formation. In his study of the history match of combustion tube runs with two different crudes, he concluded in one case that coke is always the source of fuel (Pelican) and in the other case, if non-catalytic cracking occurred, only 21% of the fuel burned was coke, the rest being the crude oil.

(ii) 10-20 kcal/mole: these could be characterized as numerical simulator values. Only two experimentally determined results fall in this range:

(a) Madgewick's 16.5 kcal/mole which was derived from fluidized bed (100-200 mesh sand) cracking of Lloydminster oil and residence times of 1-8 seconds which clearly do not fall in the time range expected for the advance of the firefront. The temperature interval used by Madgewick, 500-800°C (930-1472°F) is also at the extreme end; since activation energies are obtained from plots of ln(k) vs. 1/T which are not anticipated to be linear over all temperatures, extrapolation of his data to other temperatures may not be correct.

(b) 14.05 kcal/mole obtained by Thomas from analysis of gas samples taken using a moveable probe inserted into a combustion tube. The mathematical analysis method was complex and the result dependent on the initial guess so that confirmation of this would be desirable. The authors cite a number of other publications where the activation energies are in accord with theirs, but in fact these papers were all on LTO in the temperature range 60-120°C and applicable to full crudes since they were obtained by oxygen uptake work. These low numbers for activation energies seem quite reasonable for simulation work on spontaneous ignition in the start-up phase of a combustion drive - the experimental verification is good; they may also be useful to some extent when flame fronts are such that temperatures are low and oxygen can penetrate beyond the flame. They seem less reasonable when temperatures are high and oxygen is expected to be fully consumed in the high temperature zone.

5.3 Activation Energies - Discussion

The reasons for activation energies, as used by simulators and as determined by kinetic means, falling into two groups can be rationalized in two ways neither of which can be excluded at present. They are as follows:

5.3.1 Numerical Simulation Requirements

The low activation energies for coking in simulators may be due to the inadequacies inherent in the present kinetic models, specifically the discretization into blocks. Creation of the blocks is necessary for economic operation of the simulation program. Sufficient coke must be formed by the simulation ahead of the combustion zone to generate the energy (by combustion) to maintain advance of the front. In each time step, the temperature of each block is recalculated; this is the unique temperature that applies throughout the block. In a combustion tube, however, the temperature profile can be quite steep, e.g. in one combustion tube run, over a fifteen cm distance (one block dimension) immediately preceding the firefront the temperature increment was normally 250-300°C. Because of the exponential variation of rate with temperature, actual rate variations within the block will be very large, although the calculation procedure will of course impose a single rate. The effect will introduce a distortion reducing rates. This outcome can be compensated for in the simulator by using lower activation energies so that coke formation begins to occur much earlier and does not accelerate as much in the high temperature zone. Decreasing block sizes in steps down to the magnitude of the burning front thickness (1-2 cm) is an expensive proposition, but some preliminary work has been done at Gulf R&D to examine the relationship between grid block size, its average temperature (and hence effect on kinetically influenced events) and history matching. It was found that a decrease in block size caused peak temperatures to drop, so that coke concentrations decreased, eventually leading to cessation of combustion. The reasons for this effect are not at all clear, but may be related to the inadequate description of the cracking kinetics used in the simulator. In other words, the smaller grid block sizes used were more realistic, but the description of the cracking kinetics (e.g. activation energies) used remained unrealistic. Further study could not be pursued because of high computing costs as grid block sizes were reduced. Transfer of these programs to run on personal computers will be one way to overcome their cost of operation, and encourage more work in this area.

Before leaving this aspect, it should be mentioned that in Crookston's model, in the coke formation term expressed as:

coke rate of formation = Arrhenius term $(1 - N_c/N_{cmax})$
where N_c = coke concentration and
N_{cmax} = upper limit on coke concentration

the additional term ensures that if the coke kinetic terms involved would lead to too much fuel formation, then a limit is placed on the fuel that can be made in the simulation. This approach puts an artificial constraint on coke formation and is, in effect, also a departure from first order irreversible kinetics.

5.3.2 Matrix Catalysis of Coke Formation

Catalysis of coke formation by clay and/or fine sand may increase cracking rates and may result in lower activation energies for this step. There is no doubt that clay has an overall effect on the combustion process but quantitative measurement on the fuel formation step are sparse. The effects that clay has been demonstrated to exhibit are as follows:

1. Bousaid (17) in his combustion experiments on previously prepared coke samples found that 20% clay added to the sand reduced the activation energy for combustion from 14.5 to 11.5 kcal. In addition, fuel deposition in the presence of clay was substantially greater than in its absence. Unfortunately, not enough runs were performed to quantify these results.

2. Vossoughi (18) performed combustion tube experiments in the presence and absence of clay and silica powder. Combustion front velocities decreased when either was present and peak temperatures increased. The effect was concluded to be a surface area one and not specific catalysis by sites on the clay surface. TGA work by the same author also pointed to a catalytic effect of the clay; this aspect is discussed more fully in the section covering TGA results.

3. Fassihi (19) interpreted the results of some combustion tube runs in terms of clay causing greater fuel laydown (change in front velocity and temperature).

4. Henderson (5) found in thermal cracking experiments on Athabasca bitumen that addition of native sand/water had no effect on the rate of cracking. He was probably using a good grade sand with a small surface area/little clay thus the absence of any effect in this case may not be surprising.

5. Cormack (20) has found that clay promotes the cracking of Athabasca bitumen (after low temperature oxidation) and that less coke was formed.

6. In contrast, Phillips (21) performed closed vessel kinetics on Athabasca bitumen in the presence and absence of sand and found that there was a sharp increase in coke formed when sand was present and the activation energy for its formation was decreased by approximately ten percent.

7. Culham (4) applied both non-catalytic and catalytic mechanisms for coke formation in numerical simulations of fireflood runs on two crude oils. Both mechanisms gave reasonable history matches, but parameter sensitivity was present.

The reasons for the possible effect of clay or sand have not received detailed attention. Surface area is the most widely attributed cause, but one interesting paper (22) has shown asphaltenes and resins are adsorbed almost irreversibly onto montmorillonite, with factors affecting the adsorption including the exchangeable cation on the clay, and basic nitrogen in the petroleum fractions.

5.3.3 Numerical Simulators

Most numerical simulators in use at present are kinetic based ones; they attempt to calculate the firefront rate of advance and characteristics from the kinetics of the individual chemical and physical steps involved. As previously pointed out, the weakness of this approach is the grid block constraint and the few grid sensitivity studies that have been done have not brought the size down to the very small dimensions required for a close look at the cracking and combustion zones.

Several alternative methods have been investigated. Youngren (23) developed an implicit simulator which assumes that oxygen is consumed instantaneously in contact with the fuel, thus the rate of combustion is directlly related to the oxygen flux. This approach was adopted since in the author's opinion "quantitative data on high temperature combustion kinetics of crude oils in porous media is inadequate to allow rigorous treatment of reaction kinetics". Mobil have represented the burning zone as a moving heat source and as a "displacement pump" enhancing oil flow in their THERMS simulator which employs no kinetic data (24). To the extent that kinetic models do use real kinetic information, these alternative simulations are much more empirical in that they are dependent on combustion tube runs for their input data. For the kinetic models, the combustion tubes can be considered as a means to refine the model. Inherently though, the non-kinetic methods are less capable of further development and are probably less useful as problem solving tools.

Computing cost is the major reason for the limitation on the number of grid blocks employed in simulations, particularly in 3D cases. It will be evident that in the regions of a combustion tube or reservoir removed from the firefront, the changes occurring will be relatively small and large blocks should be satisfactory. This need for variable sized blocks has been recognized in some quarters, but the difficult part in practical incorporation is their movement with the cracking and combustion zones. To our knowledge, this has not been pursued beyond the idea stage, but if successfully completed might well represent major progress in the application of this type of numerical simulation.

6. ISOTHERMAL REACTOR WORK - EXPERIMENTAL

A schematic of the equipment used for this part of the work is provided in Figure 1. It consisted of a nitrogen supply ('oxygen-free' nitrogen from Liquid Air) with regulator (Matheson) providing an inert gas stream to the reactor. A pressure gauge (upper limit 1000 psi) and blow out disc (1000 psi) were connected to the gas feed line via tees.

The reactor was heated by a compressed air ebullated sand bath (Techne Model FB-07) in which it was almost fully immersed. The short portion which remained above the sand bed was wrapped with insulation prior to each run, to prevent thermal energy loss. For all runs except the first few, the inlet tube to the reactor for the nitrogen was led to the bottom of the reactor. Outlet tubing from the top of the reactor was heated by tape to prevent condensation of liquids in this area. Two knockout pots, one maintained at dry ice temperature, removed liquids at that point and protected the mass flow controller (Matheson Model 8240) and pressure regulator (Grove).

An alternative line by-passed the Matheson Flow Controller and could be used if the latter failed. This led to a wet test meter after the pressure regulator and acted as a back-up if the controller failed. This by-pass was never required in practice.

Two types of reactors were employed, all with 1 inch i.d. working volumes, and all built by Hulleman Industries of Burlington, Ontario. Reactor designs are provided in Figures 2 and 3. The first reactor built was slightly larger than the second and third, which were identical. Reactors were constructed from 316 stainless steel and conformed to Section VIII (Boiler and Pressure Vessel) of the Ontario Pressure Vessel Code. They were registered with the Pressure Vessel Safety Branch of the Ontario Ministry of Consumer and Commercial Relations. Each reactor was statically tested to 3800 psi by the manufacturer, and prior to use was tested by Gulf with nitrogen to a pressure of 2500 psi at ambient temperature.

The reactors came equipped with a centre thermocouple well in which were three thermocouples, located at the top, middle and bottom positions of the working volume. Thermocouples were also placed in the tape-heated upper section of the reactor which projected from the sand bath, and in the sand bath itself. These thermocouples were of Type K. Temperatures were monitored on a Yew multipoint Hybrid recorder, together with the signal from the mass flowmeter.

7. ISOTHERMAL REACTOR WORK - OPERATION

The reactors were loaded with the crude oil and matrix mixture in the inverted position with the centre thermocouple well already installed. A stainless steel mesh screen in a stainless steel holder was placed at the bottom of the reactor and the mixture added in small quantities; after each addition the material was redistributed and tamped gently with a steel rod. When the reactor was full (about 200 g material was required), a second mesh screen was inserted to maintain the artificial core in place. The seal and closure were then inserted and the closure tightened up with a torque wrench. The torque required to produce a gas-tight seal with graphite seals was usually about 40 ft.lb. The reactor was then pressure tested to about 1000 psi for leaks prior to each run.

The procedure for performing a run was to initially bring the sand bath temperature to about 50°C above the planned temperature for the run. When the desired bath temperature was attained, the reactor was connected to the gas inlet and exit tubing; once nitrogen flow and pressure (500 psi) were established, the sand bath was raised by means of the jack underneath it, until it was fully in position. The thermocouple readings were monitored at this stage, and when the reactor bottom temperature was within 50°C of the run temperature, the sand bath temperature was reduced to that planned. With the exception of some of the early runs, good agreement between the three thermocouples in the reactor was attained (usually about 1°C difference). Some of the discrepancies observed at the beginning of the work were due to defective thermocouples. A typical trace of the thermocouple temperatures through a run is provided in Figure 4.

At the completion of each run, the sand bath was lowered, and the reactor allowed to cool. At 300°C, the nitrogen supply was turned off, and complete cooldown took place overnight. When opened, the contents were removed and divided into three parts as they came out, referred to as 'top', 'middle', and 'bottom', each being weighed. A composite sample was then blended quantitatively from the three parts, and this, and samples of the 'top', 'middle', and 'bottom' were Soxhlet extracted with toluene to remove residual oil. After drying to remove solvent these samples were submitted for analyses for coke content as determined by percent carbon.

8. ISOTHERMAL REACTOR WORK - PRELIMINARY RUNS

8.1 Pressure Integrity

Reactor sealing became evident as a problem in the first few preliminary runs. Excessive torque (65 ft.lb.) had to be applied to the closure nut to obtain a gas tight seal, and this torque increased with each successive run, reaching as high as 120 ft.lb. The consequence was scoring on the metal surfaces and a further decrease in the integrity of the seal. The cause was found to be incorrect metal seals provided by the manufacturer; their hardness was greater than that of the 316 stainless steel body and closure.

The supplier subsequently provided copper rings, which did result in a gas tight seal at ambient with low applied torque, but when the reactor was immersed in the sand bath, significant leaks developed rapidly due to the different thermal expansion coefficients of the copper and stainless steel.

The problem was finallly solved by use of graphite (Graphlox) rings, which worked extremely well. Although routine replacement was necessary, no runs had to be abandoned due to pressure leaks once the switch to this type of seal was made.

8.2 Gas Flow

The initial intention in this work was to employ downwards flow of the inert gas. This was chosen following a review of flow direction in similar published work. Accordingly, the inlet gas initially was passed through a heating coil in the bath to bring it up to reactor temperature, then through a short insulated line to the top of the reactor. The first few runs with this approach showed that it posed considerable operating problems, the most important being coke production in the exit line at the bottom of the reactor due to expelled hydrocarbons. Coke also plugged the screen at the reactor bottom, and it became evident that if this mode were to be continued, modification to the reactor would be required, specifically a much larger exit diameter from the reactor to minimize the restriction caused by coke formed there. In addition, a lower oil content in the core would have been required to make this a workable technique, and the exit tubing to the first cold trap would have to be replaced after almost every run. These changes were evidently unacceptable both from an operating viewpoint, and low initial oil levels in the core would result in low coke levels which would be difficult to measure. As a consequence, gas flow as changed to upflow mode and oil content in the core was maintained at 10 percent by mass. Since even at this oil level, coke analyses turned out to be a more difficult problem than expected, the correctness of this decision was verified.

The nitrogen flow rate selected for this work was 650 cc/min, which corresponds approximately to 200 SCF/ft^2/h. For combustion tube work, this is a relatively high number, but it does correspond to gas flow rates used in a number of combustion tube runs performed on core material from this reservoir.

8.3 Materials

The Pelican crude oil employed, from N. Alberta, was a sales tank sample with less than 0.5% water. The Saskatchewan Delmas crude was a wellhead sample, which was dewatered by distillation (any crude light ends co-distilling with the water were separated and recombined with the oil phase). Inspection properties of these crudes are provided in Tables 2 and 3.

Ottawa sand (35 mesh) was the principal component of the simulated core material used in all the runs. It was used as received. The montmorillonite and illite were obtained from Ward's Natural Science Establishment, Rochester, N.Y. The kaolin was from The Pottery Supply House, Oakville, Ontario. All were dried at 800°C in a muffle furnace prior to use and passed through a 60 mesh sieve. Their surface areas (m^2/g), measured by nitrogen adsorption were found to be: kaolin - 2.6, montmorillonite - 2.4, and illite - 2.6. These areas must be regarded as approximate since they correspond to the low end of this method.

Sand and sand/clay blends with crude oil were made up by mixing the oil with the clay first, if present, to ensure coating the clay with the oil, and then adding the sand. The mixture was then rolled for several hours to ensure good homogeneity. Completed mixtures were stored in sealed bottles in a refrigerator. Crude oil content in every case was ten mass percent.

8.4 Coke Determinations by Carbon Analyses

As planned, the method used for coke determinations was by combustion analyses followed by carbon dioxide determination using the Leco Model CR-12 Carbon Determinator. Few problems were expected with this technique since extensive in-house experience showed that for coke-on-catalysts it provided repeatability at up to 2% carbon to the second decimal place. Such precision (and accuracy) was not found to be the case with samples generated in this work. Repeatability experienced on duplicate samples was poor, particularly at high (>1%) carbon levels. After some investigation, this was concluded to be due to surface coke on the matrix breaking off during sample handling. Figures 5 and 6 which are photographs taken through a microscope of synthetic core samples after individual runs demonstrate this very well; the coke and oil particles can be seen attached to the sand grains. Their easy dislodgement and loss will lead to erratic carbon analyses. No ultimate solution was found for this problem during the course of this work, and the best resolution was taken to be reducing handling of the samples to a minimum and multiple analyses of the Soxhlet-produced products from the reactor, with outlying results discarded from the averaging procedure. As a back-up set of results, the analyses on the 'top', 'middle' and 'bottom' samples were used together with the weights of these portions to calculate a further 'composite' result. Generally, the comparison of coke levels determined by this method with those from the regular composites was quite good.

9. RESULTS AND DISCUSSION - ISOTHERMAL REACTOR WORK

Sets of isothermal reactor runs were performed with ten percent by weight of Pelican crude on Ottawa sand, and 80% Ottawa sand/10% of each of the three clays kaolin, montmorillonite, and illite. One set of runs was performed with 10% Delmas crude (from Saskatchewan) on 80% sand/10% illite. Illite was the clay found to provide the largest coke level in the runs with the Pelican crude. In all, eighty-three isothermal reactor experiments were carried out.

Each set of isothermal reactor runs consisted of three runs at four temperatures between 380 and 435°C. Run times were 2, 4, and 8 hours, the latter representing the maximum time possible due to the 2-3 hours required prior to the actual operation.

Coke analyses on the composite samples from this work are summarized in Tables 4-8, and are plotted in Figures 7-11 where curves have been drawn through the data points. It can be seen that in some cases the data points do not fit on smooth curves - in these cases best estimates have been made for drawing the curves. Repeat runs have been performed where possible in attempts to improve the results but the reproducibility has been good. The sand/kaolin runs at 435°C are the most obvious instance of this where two of the three coke results lay below the corresponding points at 420°C. Coke analyses on 'top', 'middle' and 'bottom' samples from the reactor are given in Table 9.

To consider the graphical data, inspection of Figures 5-9 show the following features:

(a) Coke levels increase with time and temperature, as expected.

(b) With the exception of the sand/kaolin case, coke formation at 380°C shows what might be termed an induction time, implying formation of a pre-coke species. For the 2 and 4 hour runs, only small amounts of coke are formed, whereas this increases sharply when the reaction time is increased to eight hours. The effect appears to be consistent (including Delmas crude), and has been seen in other related work in this department. Hayashitani's (1) and Phillip's (21) studies on coking of Athabasca bitumen provided no evidence for this phenomenon.

(c) All of the three clays produced an increase in coke relative to sand alone. Clay promotes coke formation.

(d) Illite was clearly superior to the other two clays in promoting coke formation with Pelican crude. Thus no runs were performed with this mixture at 435°C with Pelican and 410°C was selected as a substitute temperature.

(e) Kaolin and montmorillonite are about equivalent in the amount of coke they produce.

(f) Comparison of the Pelican and Delmas crudes with sand/illite shows that the latter generally results in higher coke levels. Delmas crude has a lower API gravity and a higher Ramsbottom carbon than Pelican and fireflooding experience indicates that it should indeed produce more fuel.

(g) In the Pelican case, coke levels greater than 1.5% are considerably beyond fuel consumption data as measured in combustion tube runs on actual Pelican core material. Experimental fuel consumptions lie in the range 0.8-1.4 lb per 100 lb core, at crude contents similar to those used here.

(h) If fuel is expressed as percent of oil burnt, combustion tube data for Pelican cores lead to the range 12-19%, although extremes were measured above and below these numbers. In these terms the coke numbers produced here are more reasonable.

(i) At similar or identical times and temperatures, the amount of coke formed from Pelican crude with sand and illite present is substantially less than from Athabasca bitumen. This conclusion is derived by comparison of our experimental results with those of Hayashitani (1) and Phillips (21). This should be the case given the properties of Pelican crude and Athabasca bitumen. The same comparison is true for the Delmas case, although it's API gravity is similar to that of Athabasca bitumen.

(j) This part of the study shows that at the 10% level, clays have quite a significant effect on coke formation. In the TGA work described subsequently, much larger clay levels are required to produce appreciable changes to residual coke levels. This is attributed to the differences in pressure and time in the two types of experiments.

Kinetic data was obtained from the isothermal reactor work for coke formation by assuming a first order kinetic expression of the form:

$$C = P(1-e^{-kt})$$

where C is coke formed (g/100 g), P represents the sum of the coke precursors (g/100 g), k is the rate constant, and t is the time in hours. This can be arranged to the form:

$$k = (1/t)Ln(P/(P-C))$$

from which k can be calculated for each value of C at a given temperature provided that P, the amount of coke precursors is known. The best fit value of P was found to be 2.0 for the Pelican runs by determining k for the range of values for P, recalculating C for each k so found, and obtaining the best match between calculated and observed coke levels. An averaged value of k at each temperature was then obtained. These numbers are summarized in Table 10. In the case of the runs at 380°C, the rate constant for the 2 h run has been dropped in the averaging process since in each case it is a poor fit with the other two data points.

Arrhenius activation energies were obtained by plotting Ln k vs. 1/T (K) in Figures 12 and 13 for the Pelican and Delmas cases, the the Ea's are summarized in Table 11. For Pelican crude, it can be seen that the Ea's do not differ very much, indeed they are within 5 kcal of their mean which is well within the precision of the activation energy determinations. The values obtained here are also very similar to those obtained by Hayashitani and Phillips in their more detailed models of Athabasca bitumen cracking. Consequently, one must conclude that within the precision of the present technique the clays do not result in a decrease in coke activation energy, but they obviously do increase the rate constants for coke formation. In this latter aspect our results are in agreement with those obtained by Phillips; however, these workers also found that there was a decrease of approximately ten percent in activation energies caused by the clay. The simplified model we have used does not show this.

The Delmas/Illite plot for Ln k vs. 1/T in Figure 13 shows that, for the same temperature, rates of coke formation are higher than in the Pelican cases, and that the activation energy is approximately the same.

The basic purpose for undertaking this work was to determine if there was a major decrease in activation energies for coke formation caused by clays. In effect, did the Ea's come down from around 50 kcal to the values of about 25 kcal used in simulations? The answer from this work is that they do not, and Phillips' more detailed study agrees with this. Consequently, it is reasonable to conclude that further work along these lines will not be profitable in bringing simulation and experiment into agreement. Attention should therefore be turned to examination of the effect of reducing grid block sizes in simulations, as discussed in the introduction, to see if this is the source of the difference between simulation practice and chemical experience.

10. THERMOGRAVIMETRIC ANALYSES - INTRODUCTION

TGA - Thermogravimetric Analysis - is a thermal technique in which the weight of a small sample of material under study is continuously monitored as the sample is heated. If the temperature of the sample is increased as a linear function of time, the method is referred to as non-isothermal TGA. This variation has been widely used in the quantitative study of the thermal decomposition of both organic and inorganic materials and the theory has been well developed. Among the advantages of the technique as currently practiced are the automated equipment now available with electronic data storage, and the small amount of operator time required per run. With these apparent cost effectiveness advantages, it is not surprising to see the methodology applied to problems in the Fossil Fuel Energy area. The applications so far have been to cases where particular developing technologies involve thermal cracking steps. Thus coal pyrolysis, shale oil retorting, coking process development, and the recovery of crude oils by in situ combustion, have all been studied to a greater or lesser extent by this technique.

Voussoughi et al. (27) used a combination of TGA and DSC (Differential Scanning Calorimetry) to study the effect of kaolinite clay on crude oil combustion, in the presence of a flow of air. Their TGA thermograms showed three distinct regions - one attributed to distillation, and two to combustion/cracking. Kaolin clay was found to change the shape of the TGA/DSC thermograms and significantly reduce the activation energies of the combustion/cracking steps relative to those for crude oil alone or crude oil/sand mixtures. Under the conditions used in this work, combustion and cracking are occurring almost simultaneously. This is a different sequence from that generally regarded as taking place in the process where cracking precedes combustion and occurs in oxygen-free conditions ahead of the firefront, with the coke being burned, not the volatile products of combustion.

Jha (28) obtained TGA's under nitrogen and helium for two Lloydminster heavy oil core samples, calculating activation energies for four temperature spans - 50-340°C, 340-475°C, 475-550°C and 550-800°C - based on single runs using the calculation method due to Coates (29). Bearing in mind that the oil present was a full range crude, activation energies in all cases increased over the first three temperature ranges, final values being 36-50 kcal/mole. Activation energies for the final temperature range were 14-22 kcal/mole, and represent those for the slow mass loss from the coke formed due either to loss of volatiles or slow oxidation due to traces of oxygen in the inert gas supply.

Hardy (30) has used DSC to estimate ignition temperatures of crude oil, and heat availability in core samples from a combustion pilot, and found that increased kaolin levels caused a shift of the exotherm to lower temperatures. In addition, samples taken at decreasing distances from the firefront of an interrupted combustion tube run demonstrated loss of volatile components as the temperature increased. This type of change is to be expected. Cities Service workers (31) have also applied TGA to core samples taken from evaluation wells following completion of a combustion pilot program. Variation in the thermograms was used to determine whether the reservoir in the regions from which the core samples were taken had been exposed to high temperatures or not. This means of determining the aerial and vertical sweep of the fireflood is a very useful application of TGA, particularly as it probably covers a temperature range beyond the scope studied by changes in mineral composition.

Rajeshwar (32) obtained TGA's and DSC's under nitrogen for four oil sand samples - N.W. Asphalt Ridge, P.R. Spring, and Circle Cliffs (all in Utah) and Athabasca, noting that in each case weight loss proceeded in two stages, the first of which is probably distillative loss of unaltered volatile components, and the second is due to thermal cracking of the residual material. He also noted that at a heating rate of 50°C/min, the residual 'coke' was larger than at lower heating rates, although runs at 2-20°C/min showed no variation in amount of coke. It will be seen that this dependency between coke level and heating rates is the opposite to that experienced in this work.

If volatilization of distillable materials (b.p. range 100-500°C) is accepted as occurring in advance of the firefront, then cracking of the residual 500°C+ may be the kinetically significant step in fuel laydown. R.C. Schucker of Exxon R&D has pointed out in two papers (15,33) that a constant activation energy does not hold for residual cracking, rather there is a continuum of activation energies which increase steadily with extent of conversion.

Thus, his non-isothermal TGA work on a whole Arab 565°C+ resid found that activation energies increased from 36 to 60 kcal/mole as conversion went from 20 to 90% reaction; furthermore, these values varied with the asphaltene, polar, aromatic, and saturate components of the residue. As he points out, his results bring residue cracking into line with the coal pyrolysis studies of Anthony and Howard (34), and modelling work on any of these systems would be incorrect if a single activation energy is employed. These conclusions have an obvious impact on the heavy oil cracking step used in modelling in situ combustion runs.

Finally, in this review of applications of TGA to residuum cracking and in situ combustion, an internal Gulf project (35) found in preliminary work that added kaolin caused an increase in coke formed during TGA work and also appeared to reduce overall the range of activation energies determined for a sample of Pelican crude resid (504°C+). Fine silica powder was also found to have a similar but lesser effect. Accordingly, the TGA technique was considered to hold some promise as a means to study the effect of clay on the magnitude of fuel laydown, and its influence in the catalysis of cracking reactions.

The work reported here is the most extensive one undertaken to date of the effect of clay and sand on coke yields and activation energies from the thermal decomposition of a petroleum residue.

11. TGA - EXPERIMENTAL

11.1 Pelican 504°C+ Residue

The Pelican vacuum residue employed in this work was prepared by TBP distillation of a sample of Pelican sales tank oil to a FBP of 504°C. Properties of this vacuum residuum are given in Table 12. The GCD (without internal calibration) shows that about 13% of the distillable material in this sample boils below 504°C with the 5% point at 413°C. At this temperature (413°C) in the TGA's the extent of reaction was considerably more than 5%, generally in the 25-50% range, thus decomposition rather than distillation was the main contributor to mass loss, during the TGA studies.

11.2 TGA Analyses

Experimental thermal analysis work was carried out using a Omnitherm 25TG950 thermogravimetric module supervised by a DuPont 1090 Thermal Analyser based system. The 1090 base unit performs module programming and control as well as data acquisition and analysis using a DuPont 1091 dual disk memory unit.

To exclude oxygen from the sample as the temperature was raised from ambient to 600°C, the standard balance bell jar was replaced by one with a nitrogen gas inlet located furthest from the sample. In addition, the electronics cable inlet to the balance mechanism was thoroughly sealed with silicon sealant. Early work showed that slow mass loss at sample temperatures greater than 525°C were due to oxidation of the coke by trace amounts of oxygen in the nitrogen. Accordingly, any oxygen present was removed from the nitrogen supply by passing it through two oxygen scrubbers, one being an indicating oxygen trap (available from Chromatographic Specialties, Brockville, Ont., Part #'s C36060 and C36070). For coke yield determination at the conclusion of each run, the gas was switched automatically from nitrogen to air.

Sample sizes for thermogravimetric kinetic analyses were maintained as constant as possible, in the general range 25-28 mg. Excessive variation in sample size resulted in erratic results. As well, too large a sample was found to result in mechanical interaction between the sample and the thermocouple, due to formation of an expanded carbon 'honeycomb' structure.

For each sample type, TGA runs were performed at four heating rates - 5, 10, 15, and 20°C/min. Including the TGA work directed at coke devolatilization this study involved approximately 100 individual runs.

11.3 Calculation of Activation Energies

Two methods were employed. The first used the DuPont TGA Decomposition Kinetics Program which calculates activation energies by the method of Flynn and Wall (36). This assumes first order kinetics and requires that runs be performed at several heating rates. A brief outline is as follows:

From the basic TGA equation: $dC/dT=(A/B)*f(C)*e^{E/RT}$

where B = heating rate, C/sec, and f(C) some function of the degree of conversion, D; by integration we get:

$$dF(C)=dC/f(C)=(A/B)*e^{E/RT}*dt$$

$$=AE/BR\ (e^{E/RT})/(E/RT)+(e\ x/x)dx$$

where x=E/RT

$$=AE/BR\ p\ (E/RT)$$

$$\log F(C) = \log (AE/R) - \log B + \log p(E/RT)$$

$$= \log (AE/R) - \log B -2.315 - 0.457E/RT$$

Differentiating at constant degree of conversion we get:

$$d(\log B)/d(1/T) = (0.457/R)E$$

Consequently, for TGA runs at different heating rates (B) plotting log B vs. 1/T should give a straight line of slope 0.457E/R.

The second method was that due to Friedman (37) which also requires that runs be performed at several heating rates. From the same basic equation used in the previous section, but incorporating t=time, the following result can be obtained:

$$\ln((1/V_o)*(dV/dt)) = \ln A + n*\ln(1-V/V_o) - E/RT$$

At constant degrees of conversion, i.e. V/Vo, a plot of the LHS vs. 1/T gives a line with slope E/R. For these calculations, for each run Vo was taken as the mass loss to constant mass (in nitrogen, usually to 525°C) and dV/dt and T calculated at each 10% interval of conversion by linear interpolation. Again for each of these conversion levels, a least squares calculation of the left hand side of the equation above versus the inverse of the absolute temperature provided the slope and intercept of the line, from which the activation energy could be calculated. Correlation coefficients for the four points involved usually equalled or exceeded 0.99. For calculation of dV/dT, T and 1/T at each point, Visicalc templates were found to be the most time saving methods.

Examples of the output from these are given in Figures 14 and 15. A third method, that due to Shih and Sohn (38), was briefly employed. This is similar to that developed by Coats and Redfern (39) but is a little more sophisticated. The form of the equation is:

$$\ln(-B*\ln(1-V/V_o)/RT^2)-\ln(1-2RT/E) = \ln(A/E) - E/RT$$

Values of E and A are obtained by repeated least squares fits of the LHS against the RHS starting with some initial value of E which is updated in each cycle. Application of this method in early work found that the activation energies obtained were virtually identical to those obtained by Friedman's method. As a consequence this form was not pursued further.

11.4 Sample Preparation

Samples for the TGA work were made up by adding a preweighed quantity of the Pelican Resid, dissolved in toluene, to a known quantity of the clay sample in a 1-L flask. When well mixed, the sand required was added and the solvent subsequently removed in a rotary evaporator. The mixture was then transferred from the flask to a bottle, and stored until use in a refrigerator. For the TGA runs with Pelican Resid alone, a small sample was transferred to the TGA pan by use of a heated pipette.

12. TGA WORK - DISCUSSION OF RESULTS

12.1 Coke Formation

The amount of coke formed with each type of support for the Pelican Resid is summarized in Table 13. The samples used were Pelican Resid with no support, the Resid on sand alone, and on each of 10, 45 and 90% of the clays, kaolin, montmorillonite and illite. Coke levels are plotted against percent clay in the matrices in Figure 14. In calculating these numbers, the coke mass was defined as the difference between the sample weights at 525°C and after combustion was complete (usually above 650°C). This coke was then calculated as a percent of the total mass loss between 200°C and 700°C. In a number of cases, duplicate sets of runs were performed; these results are provided in Table 14 where it can be seen that duplicates generally agree within 2 percent absolute. Averaged results then will usually agree to within plus or minus one percent.

The following conclusions can be drawn from the data in Table 13 and Figure 16:

1. Pelican Resid, when heated without any sand or clay present, gives the least amount of coke of any of the samples used.

2. As heating rate is increased in the TGA runs, the percent of the crude converted to coke decreases.

3. Coke formation increases with the amount of any clay present.

4. Clays cause more coke to be formed than sand alone.

5. Under the TGA conditions, each of the three clays, at any level, result in about the same amount of coke being formed. In other words, the TGA technique does not discriminate between the clays, in terms of coke promotion, as the isothermal reactor work does.

6. There is little, if any, difference between the amount of coke formed when clay is present at the 10% level or absent entirely.

Overall, it can be seen that while TGA does mimic to an extent the isothermal reactor work, much higher clay levels are required to secure an appreciable effect in terms of coke formed. There are two obvious reasons for this. First, TGA studies are performed at atmospheric pressure and involve use of very small samples, thus escape of volatiles which might otherwise contribute to coke takes place. Secondly, heating rates in TGA work are much more rapid than in a reservoir and proceed to 'completion' in a much shorter time frame. It would be of interest to conduct further work along these lines at heating rates of about one degree per minute to determine if discrimination between the clays does then become apparent. A further contributing reason is that the TGA samples are missing the gas oil, middle distillate and naphtha fractions which can be expected to contribute to coke formation in that order.

The effect of heating rate on coke formed and the postulated effect of pressure are not new observations. In commercial coking operations, relatively small increases in the pressure have been found to promote an increase in the amount of coke formed (40). In coal pyrolyses, liquid yields decrease as pressure increases (41). The same effect of pressure has been found in the pyrolysis of bituminous coal, and this paper also finds that high heating rates lead to increased yields of volatiles (42).

It is relevant to this work that in the coal studies (42), two possible reasons are given for the effect of heating rate on coke levels. One, as mentioned before, is that reactive volatiles are lost more rapidly at high heating rates. The second interesting suggestion is that mass transfer plays a significant role and it was found that liquid yields exhibited a smooth relationship with particle size, the smaller particles giving the higher yields. The argument here was that with large particles, the diffusional escape of volatiles was retarded, thereby increasing the extent of secondary reactions.

This second argument probably does not apply to the cases dealt with in this work. Samples containing clay and used in the TGA studies were light in colour and at least to the eye the Pelican Resid was not immediately obvious - thus the coating of the resid on the particles was relatively thin. This is in contrast to the Resid on the sand with no clay present - the colour in this case was black, and the coated sand grains were adhered together by the bituminous material present. In this latter case one would expect mass transfer effects to be more significant where a thicker layer of resid is present; however, coke yields with sand alone present were at the low end of the scale, consequently diffusional effects did not predominate. This must also be true when TGA runs were performed with Pelican Resid by itself - coke yields were at their lowest values. It is possible that mass transfer does play a larger role in coal pyrolyses due to the higher initial viscosities of the medium being dealt with.

TGA work at variable applied pressures would be of considerable interest to determine if a better match between the isothermal reactor work and the present TGA studies could be obtained. The greatest benefit would of course be the opportunity to obtain results closer to reality and gain the benefits that TGA offers of rapid data gathering and hopefully cost effectiveness. An experimental high pressure TGA system has been described (43) and a few results at variable pressure outlined (44). From the figures in Ref. 44 the evident result of an increase in pressure of inert gas is larger amounts of residual coke. An investigation along these lines would be worthwhile and indeed essential to develop TGA such that its results could be applied directly to the in situ combustion fuel problem. High pressure TGA equipment which could be used to tackle this problem is now becoming commercially available (45), but may not yet be applicable to non-isothermal TGA.

There are good practical reasons for such a study as well. The consequence of both these factors is that excessive fuel consumption could result from either too high an applied air injection pressure or from a firefront which moves too slowly through the formation. High fuel consumption translates into high air injection rates. Since compression costs represent a major portion of the expenses in operating a fireflood, any process variables which may lead to unnecessary costs is worth further investigation.

12.2 Activation Energies

Activation energies were determined for all the runs described in the previous section. They were obtained by both the DuPont Kinetics Program and calculated by Friedman's method, both methods using the results from the four runs in each case at different heating rates. Those by the DuPont method are given in Table 15 in increments of 10% of reaction and extend only to 50% completion, a restriction of the software. Those calculated by Friedman's method have been taken to 90% completion, again at each 10% increment. The latter are presented in Tables 16-28. Also provided in these tables are the correlation coefficients for the least squares lines through each set of four points and the intercept on the y-axis. Experience showed that correlation coefficients consistently below 0.98 resulted in doubtful values of the activation energies.

Final Ea values by Friedman's method for the Pelican Resid on the various types of supports are summarized in Table 29, and are plotted in Figures 17-21. These plots show that there exists, not surprisingly, some scatter in the data, and 'smoothed' curves through the points. These are replotted in Figures 22-24 with the corresponding numbers provided in Table 30.

Corresponding Flynn and Wall activation energies from the DuPont Kinetics program are plotted in Figures 25-27. The values obtained by this method are in general similar to but not identical with those calculated according to Friedman's procedure. With the differences being minor, comparison of the sets, each calculated by the two methods, show the same trends. Thus we are dealing with a reasonably consistent set of results.

From these results the following conclusions can be drawn:

1. Activation energies for thermal decomposition of all the samples increase with extent of reaction.

2. Of the samples here, Pelican Resid by itself exhibits the highest range of activation energies.

3. As clay content of the inorganic matrix increases, activation energies generally decrease.

4. Samples with the highest clay content in each set resulted in the lowest activation energies.

5. All three clays have very similar effects, i.e. the results do not distinguish between the different types of clays.

6. The trend of the activation energies with percent reaction is an asymptotic approach to a maximum value.

7. The results provide strong evidence that clays do indeed catalyze the thermal decomposition of heavy ends.

The pattern of increasing Ea's with extent of reaction is in accord with Schucker's results on a Heavy Arabian Resid, also obtained by TGA on similar sized samples with results identically calculated. In his work he not only examined the resid itself, but also the asphaltene, polar, aromatic and saturate fractions. The Ea's obtained are plotted in Figure 28. It can be seen that in the Heavy Arabian case, the shape of the curves are different from those obtained here, increasing fairly sharply towards the final extent of reaction whereas the trend in the Pelican samples is to increase more slowly as completion of reaction is approached. The range of Ea's calculated for both cases (no clay or sand present) are reasonably similar. In the absence of any other information, we can only attribute the difference in these curves to compositional differences between the two types of crudes.

Both this work and Schucker's point out very clearly that a single activation energy for the thermal decomposition of heavy ends does not correspond to reality. Rather, a distribution of Ea's is required to provide a good model for the thermal cracking reaction. In addition, this distribution shifts to lower values when clay is present. It should be carefully noted here that the distribution of activation energies refers to the thermal cracking reaction with the ultimate formation, in the case of any given sample, of a fixed quantity of coke. The TGA work does not measure the rate of formation of coke as such, rather the loss of volatiles.

With respect to modelling of in situ combustion, most models are directed towards calculation of coke levels available for combustion. However, most models also factor into account combustion of 'heavy oil' components, so that coke is not the sole source of energy. The TGA approach leads to a different type of model in which the fuel is not coke plus some quantity of heavy material, instead the fuel becomes to be defined as that material remaining behind after exposure of the reservoir increment to a specified temperature/time profile. Further development of this model type is beyond the scope of this work, however.

In the comparisons of activation energies made so far, no mention has been made of the Pelican/Sand case (no clay present). The results with this case appear anomalous, in that the Ea's were higher than any of the other instances. Experimentally, it was also the most difficult sample to work with; the sand grains adhered tightly together due to the excess resid on the surface (relative to the other samples with clay present). The correlation coefficients for the activation energy plots were by far the worst. Activation energies by the DuPont procedure are less than those for the Resid with no support present. An additional set of runs were performed - the data are provided in Table 31 but there was essentially no change in either the activation energies or the correlation coefficients, the latter remaining poorer than expected. Table 32 demonstrates the duplication obtained with the 10% montmorillonite case and it can also be seen to be quite good. Overall, to have optimum confidence in results from this type of work, it would be best to perform the TGA at six or seven different heating rates.

In terms of expected effects, the results with the sand indicate the Ea's may be higher than for those samples which contain clay. It is less easy to explain why they are higher than the Pelican Resid case itself. The poorer correlation coefficients does not add confidence to any interpretation which might be offered to explain the differences. More experimental evidence is what is really required, with the vacuum resid of an additional oil and a similar set of inorganic matrices to work with.

13. COKE DEVOLATILIZATION

13.1 Introduction

Coke formation rates are currently assumed to follow Arrhenius' expressions and a stoichiometry. Thus, an increase in temperature should increase the amount of coke formed. However, it is also known that 'fuel' yields and H/C ratio decrease with increasing temperature thus there is a possibility that simple models are not correct.

Bousaid and Ramey (17) generated 'coke' or 'fuel' (both terms were used) at a set of different temperatures by performing a series of coking runs on thin stationary beds of sand containing oil. They noted that the fuel remaining on the sand approached in appearance that of a solid 'coke-like' substance as the coking temperature increased. The material became harder and darkened as the production temperature increased. As coking temperature increased, fuel deposition decreased and was accompanied by a decrease in H/C ratio which eventually became zero; this implies that low temperature coke loses hydrogen rich-volatiles (as small hydrocarbon molecules) to form eventually coke which is composed only of carbon and whatever minor non-volatile heteroatoms remain bonded to carbon. If the fuel is other than coke alone, the data also fit a situation where the heavy ends are undergoing increasing cracking (with loss of low molecular weight hydrocarbons) as the temperature increases.

Alexander, Martin, and Dew (46) conducted an extensive study of fuel availability using 'fire-flood pots'. One of the sets of runs they undertook involved varying only the maximum combustion temperature, with measurement of fuel consumed by carbon dioxide formed. They found that as temperature increased from 150 to 600°C, fuel available increased almost linearly as far as 350°C and declined there after, reaching a plateau of about 30% less at 425°C. Apparent H/C ratio of the fuel was found to decrease steadily with increasing combustion temperature.

These factors were recently considered to have an impact on combustion tube work by Gulf. Comparison of several combustion tube runs undertaken on similar core materials revealed that the run in which the combustion temperature was 100°C higher consumed noticeably less fuel. This decrease in fuel consumption was attributed to pyrolytic degradation of coke formed as a lower temperature to a one of lower hydrogen content.

13.2 Results and Discussion

The original intention was to examine coke devolatilization using the isothermal reactor, but experience with this unit showed it to be more time-consuming and coke analytical results less precise than expected. Accordingly, TGA was selected as the technique to be employed, and samples of sand/clay/coke from eleven isothermal reactor runs were chosen. These were either composites or were taken from 'bottom' samples in which coke percentages were higher than in the composites. All these samples had, of course, been toluene extracted so that 'coke', as defined throughout this report, was being studied. The samples used, the isothermal reactor runs from which they were taken, and the results are summarized in Table 33. All samples were run at a heating rate of 15°C/min and six were also run at 5°C/min. Weight losses were calculated for the intervals 400-525°C and 400-600°C (combustion of remaining coke was initiated at about 610°C by switching the gas flow from nitrogen to air). Thermograms for these runs and the accompanying data sheets are given in Appendix II.

The data in the % Devolatilization columns of Table 33 show that weight loss from the coke is not particularly significant - only in two cases does loss exceed 5% in the 400-525°C interval and between 400 and 600°C the mass loss is usually less than 10%. Thus coke laid down at approximately 400°C does not undergo any appreciable loss when further heated to 600°C. These numbers can be compared with those of Bousaid and Ramey's in Table 34 for fuel deposition vs. temperature, where over the temperature interval 150-480°C 'fuel deposition' decreases by a factor of ten. From our results, this is evidently not due to thermal cracking of coke, but results from thermal cracking of the crude oil itself.

Since the mass loss observed in this work is very minor, a kinetic study involving a series of runs at different heating rates was not worthwhile. We have however determined activation energies for mass loss in several of the samples studied. These were performed by the method used by Jha in his investigation of Saskatchewan cores to TGA. Calculated Ea's for our samples in the temperature range 400-600°C were all less than 1 kcal/mole. These are almost certainly too small to be due to a thermal cracking reaction. Accordingly it seems more likely that the losses seen are due either to escape of small amounts of volatiles trapped within the coke or is caused by slow oxidation of the coke by traces of oxygen remaining in the sweep gas. The main point, however, remains that losses are extremely small from the coke, and do not have to be considered in modelling the combustion process.

14. ACKNOWLEDGEMENTS

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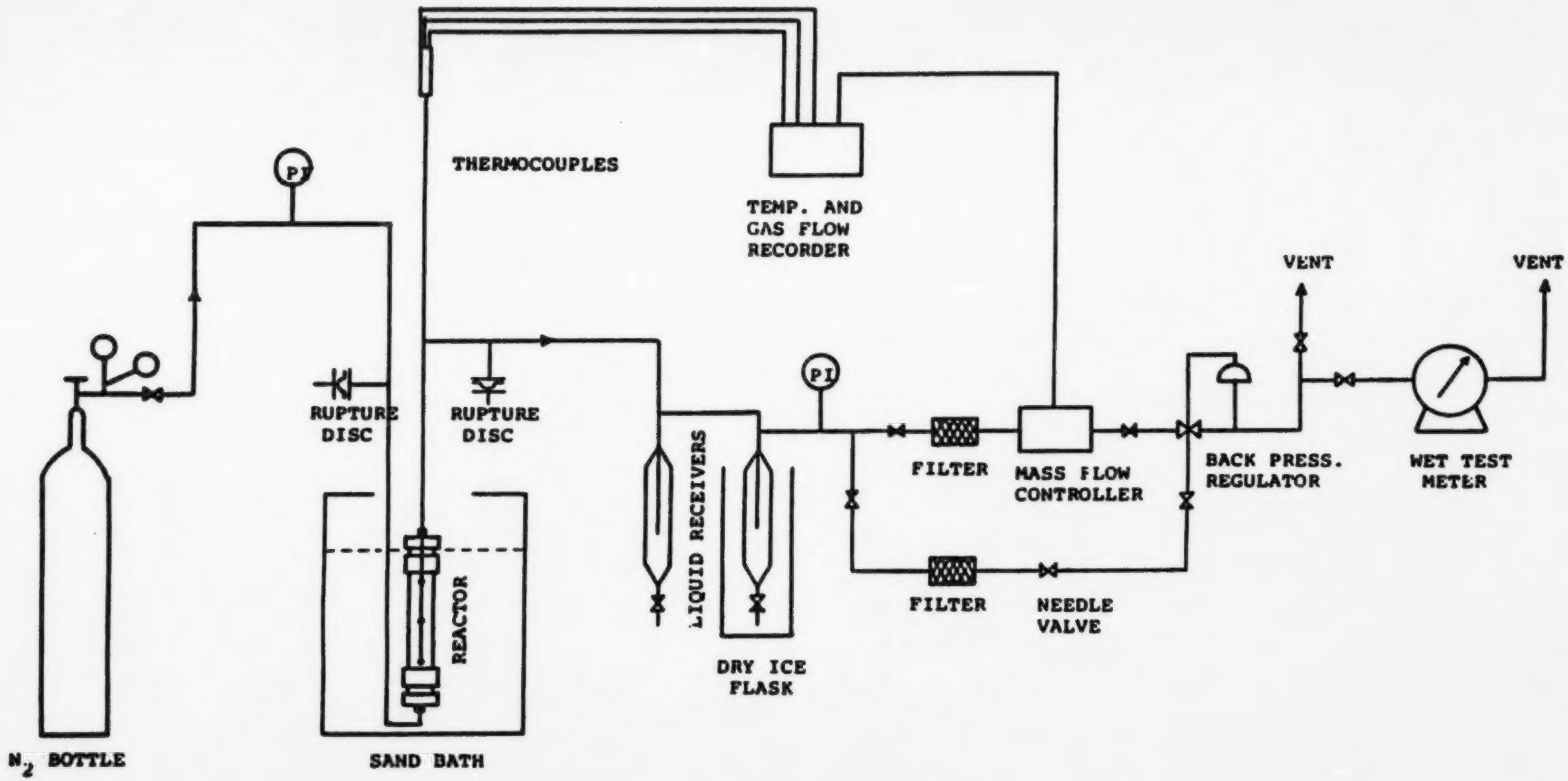
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TEMP. AND GAS FLOW RECORDER
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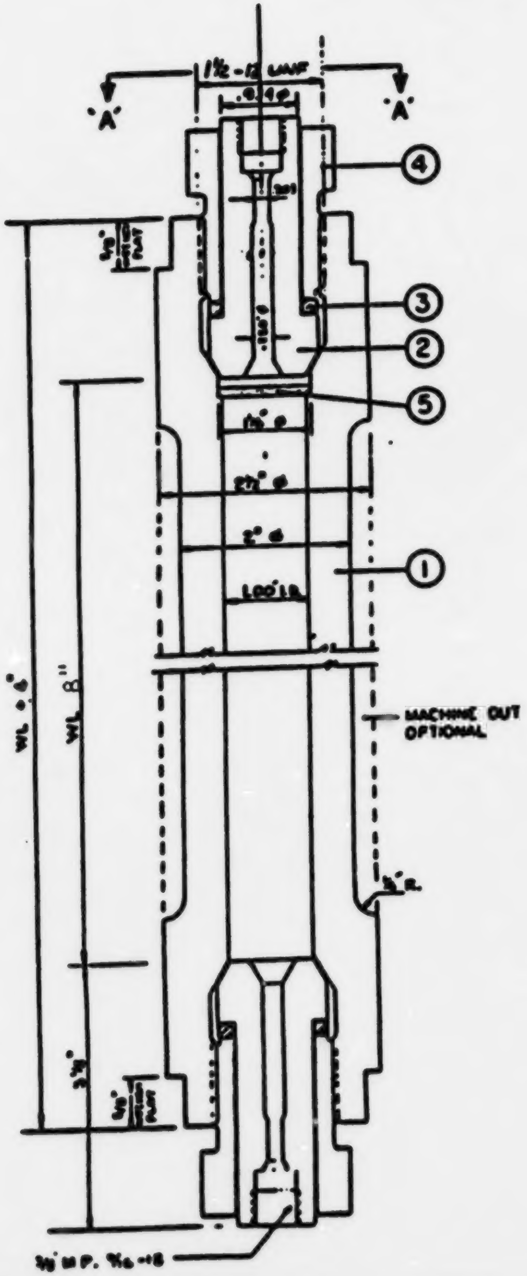
BACK PRESS. REGULATOR
WET TEST METER
REACTOR
FILTER
NEEDLE VALVE
DRY ICE FLASK
N2 BOTTLE
SAND BATH

FIGURE 1

FIGURE 2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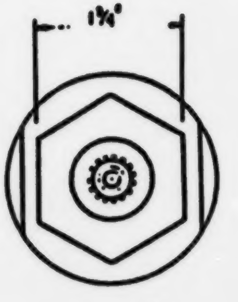

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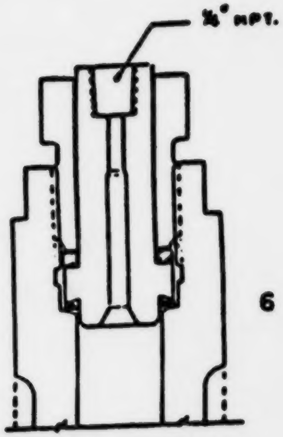
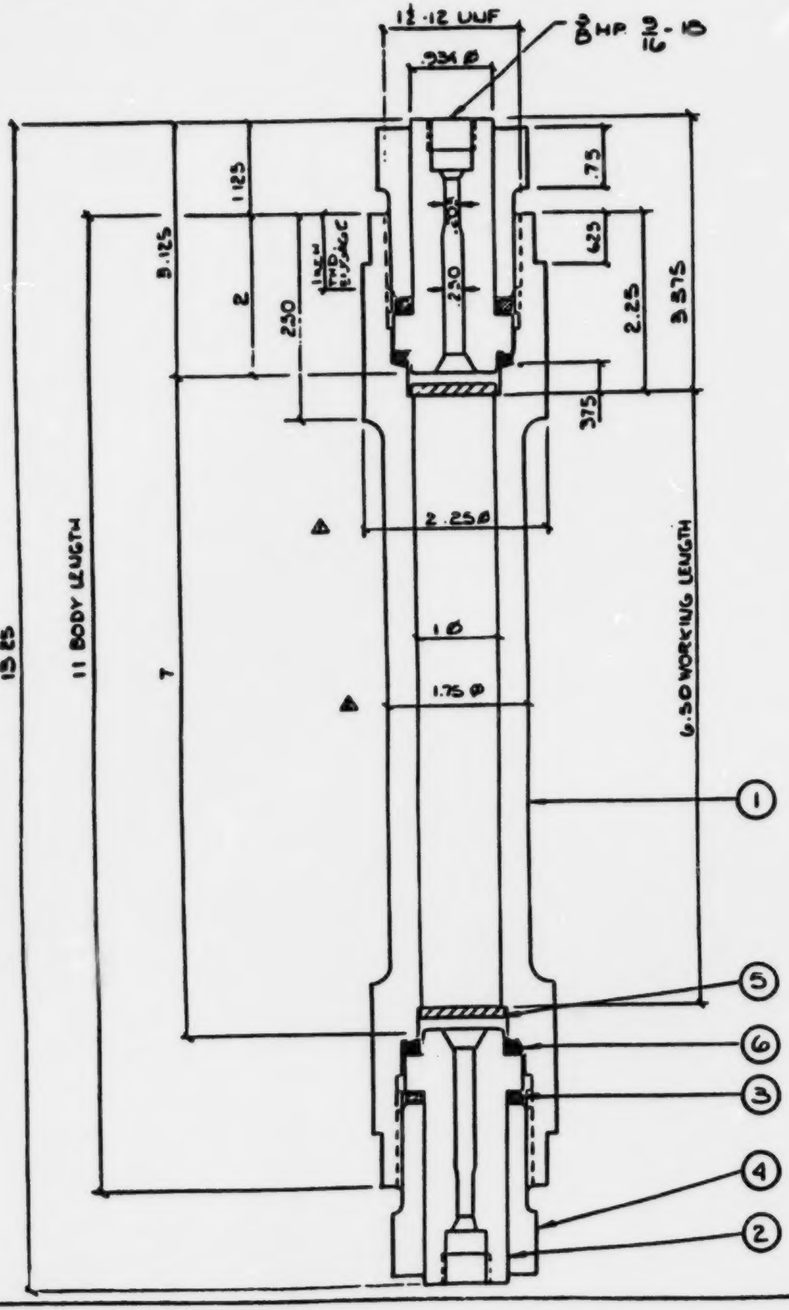


ALTERNATE DETAIL
FOR
GASKET SEAL

MAX. TORQUE REQ'D. 20 FT. lbs.
MAX. ALLOW W.P. 11,000 PSI @ 100°F
MAX. ALLOW W.P. 2,500 PSI @ 1200°F

MATERIAL LIST				
ITEM	DESCRIPTION		MAT'L	QTY.
1	BODY	PE01-1316	316 SS	1
2	COVER	PE01-2316	316 SS	2
3	BEARING WASHER	PE01-3796	A-286	2
4	MAIN NUT	PE01-4304	304 SS	2
5	SCREEN (OPTIONAL)	PE01-5	SS	
6	GASKET	PE01-618S	18-8SS	
6a	GASKET	PE01-6a-C	COPPER	

FIGURE 3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.250
.250
2.25 Ø
1.0
1.75 Ø
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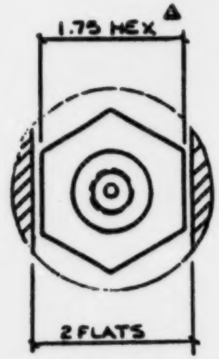
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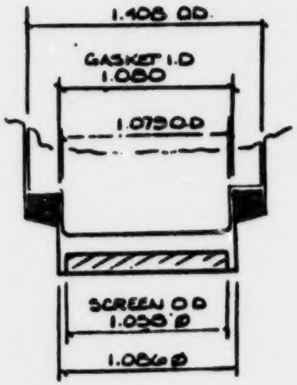
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GASKET I.D
1.080
1.0790 O.D
SCREEN O.D
1.030 Ø
1.086 Ø

MAXIMUM OPERATING CONDITIONS
PRESSURE : 1000 PSI
TEMPERATURE : 1200 °F
HYDROSTATIC : 3810 PSI @ 70 °F
PRESS TEST

MATERIAL LIST

ITEM	DESCRIPTION	MAT'L	QTY
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2	COVER	316 SS	2
3	THRUST WASHER	RA 330	2
4	MAIN NUT	RA 330	2
5	SCREEN	SS	2
6	GASKET	GRAPHITE	2

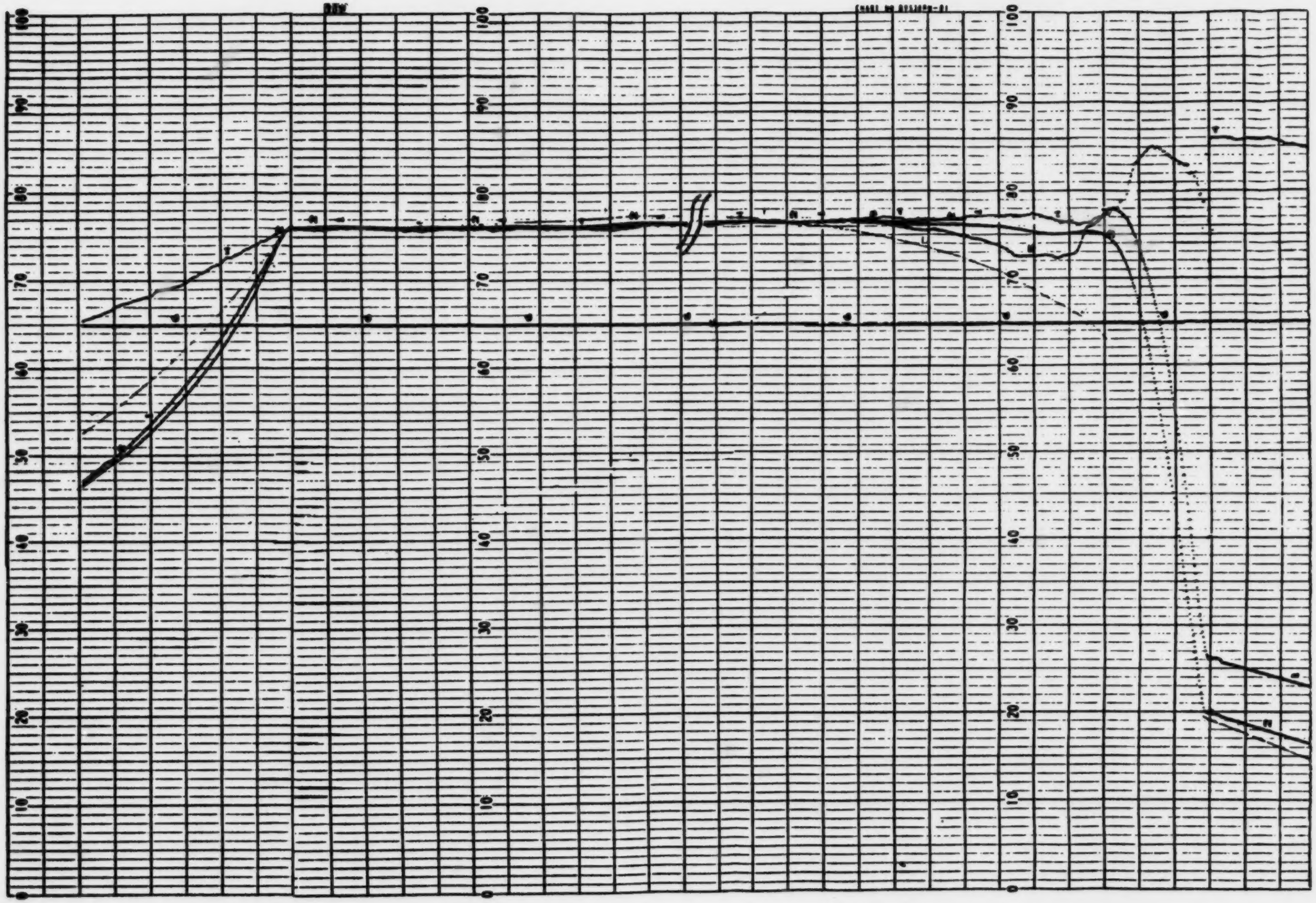

FIGURE 4

TYPICAL THERMOCOUPLE TRACES FOR ISOTHERMAL

REACTOR RUNS

FIGURE 5: SYNTHETIC CORE FROM RUNS #33 AND 35

5A: Run #33: Material from Bottom Section - extracted

5B: Run # 35: Material from Bottom Section - not extracted

FIGURE 6: SYNTHETIC CORE FROM RUNS #35 AND 36

6A: Run #35: Material from Bottom Section - not extracted

6B: Run # 36: Material from Bottom Section - not extracted

FIGURE 7: PELICAN/SAND RUNS - COKE LEVELS

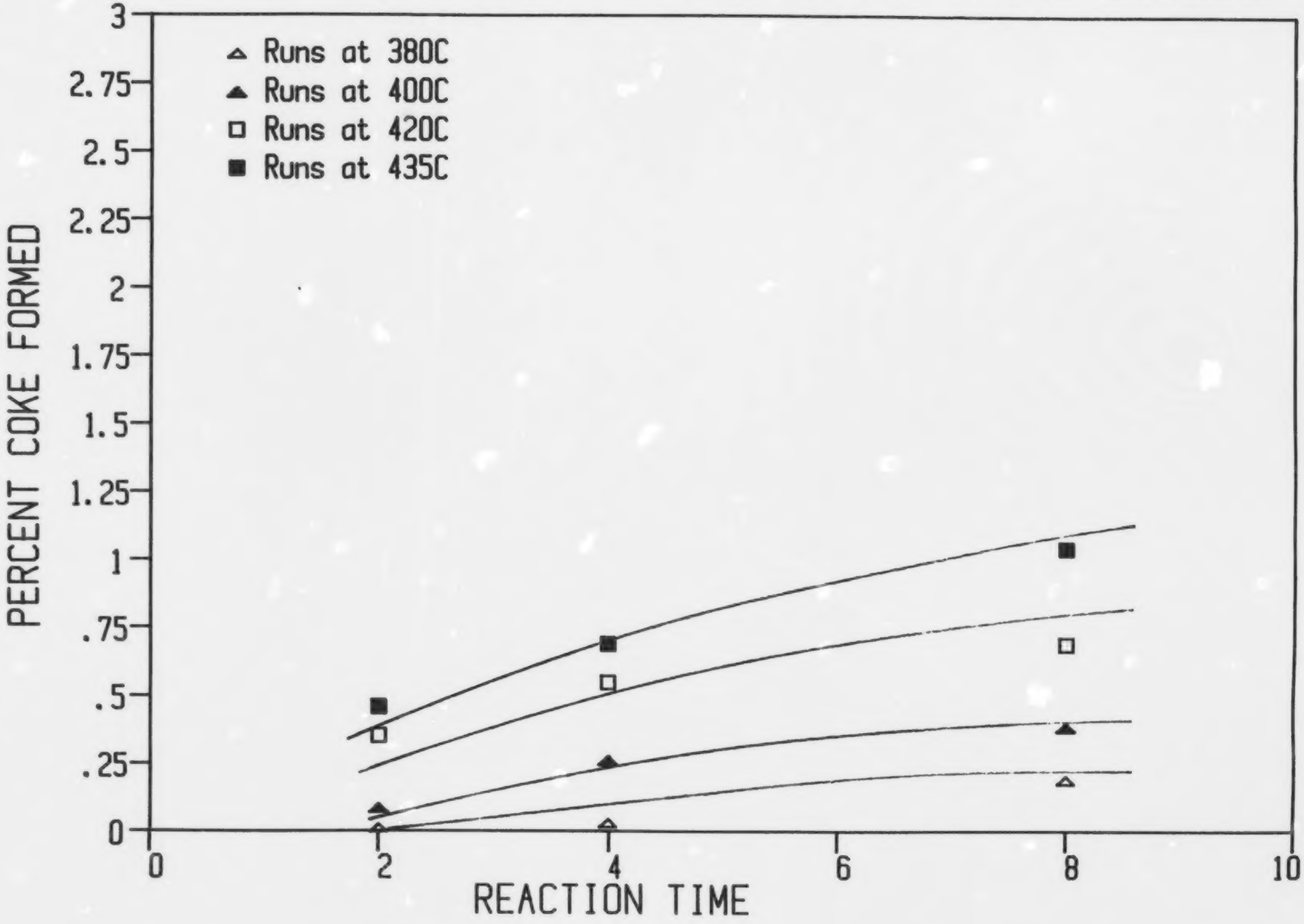

FIGURE 8: PELICAN/KAOLIN/SAND RUNS - COKE LEVELS

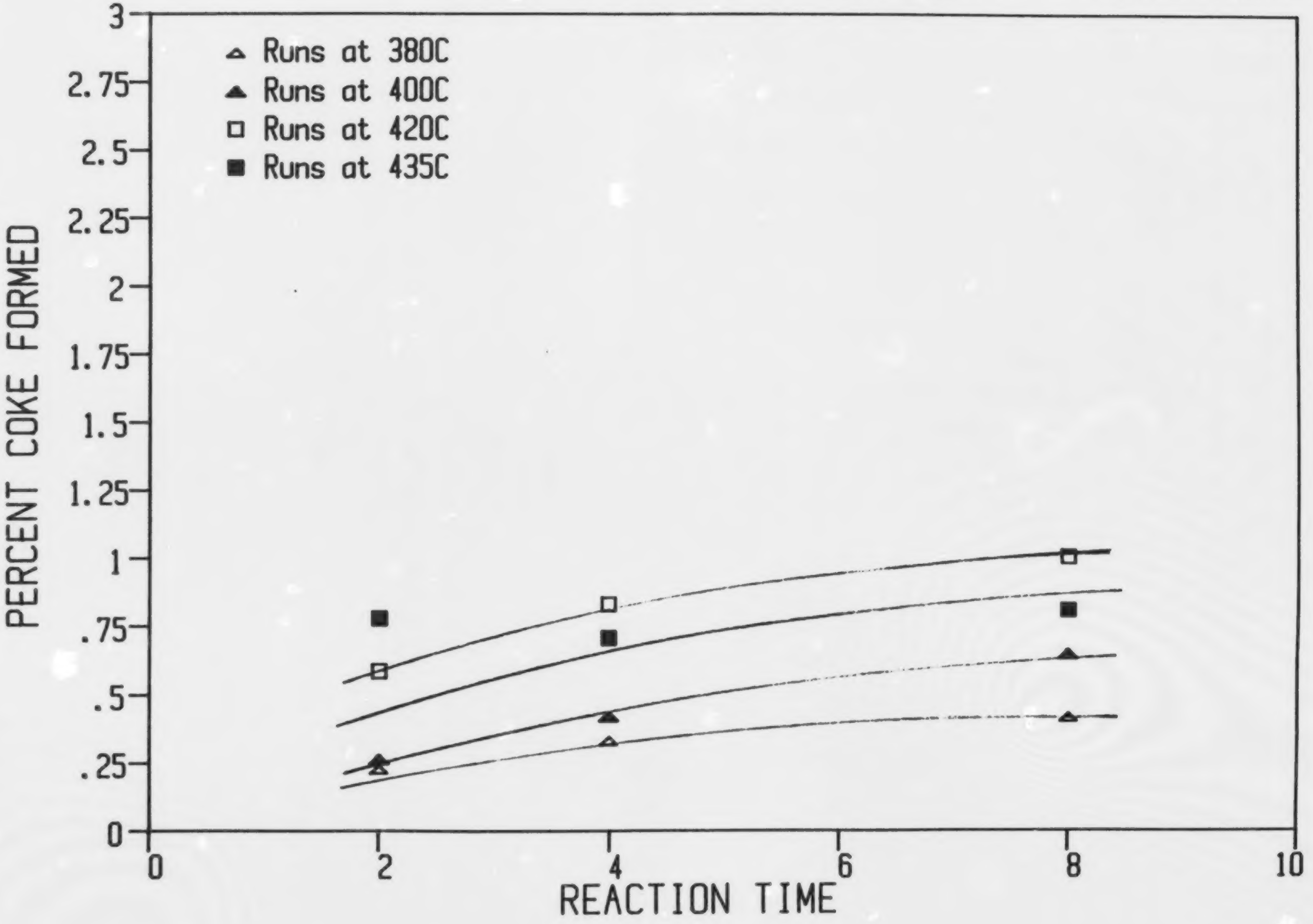

FIGURE 9: PELICAN/MONTMORILLONITE/SAND RUNS

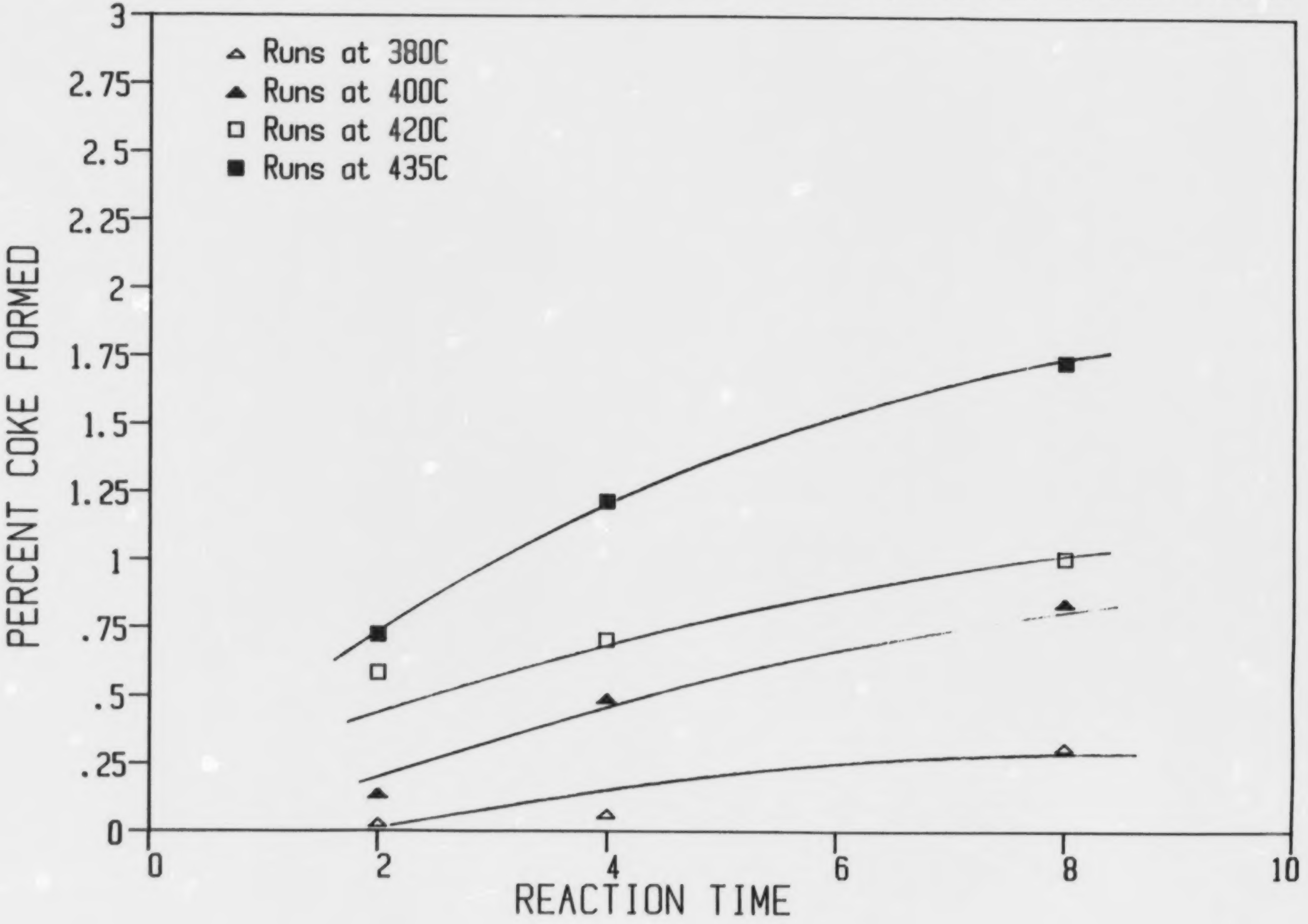

FIGURE 10: PELICAN/ILLITE/SAND RUNS-COKE LEVELS

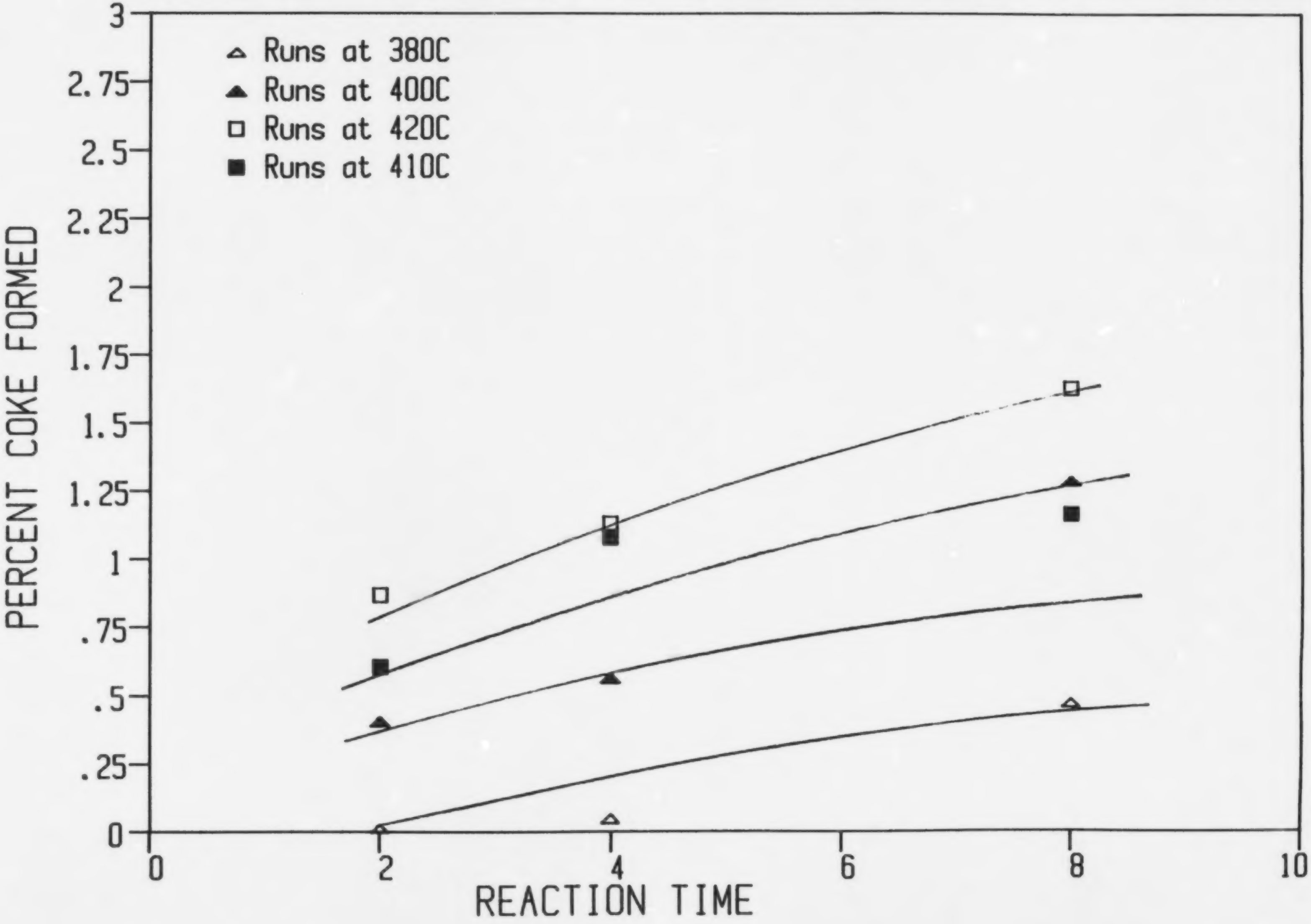

FIGURE 11: DELMAS/ILLITE SAND RUNS - COKE LEVELS

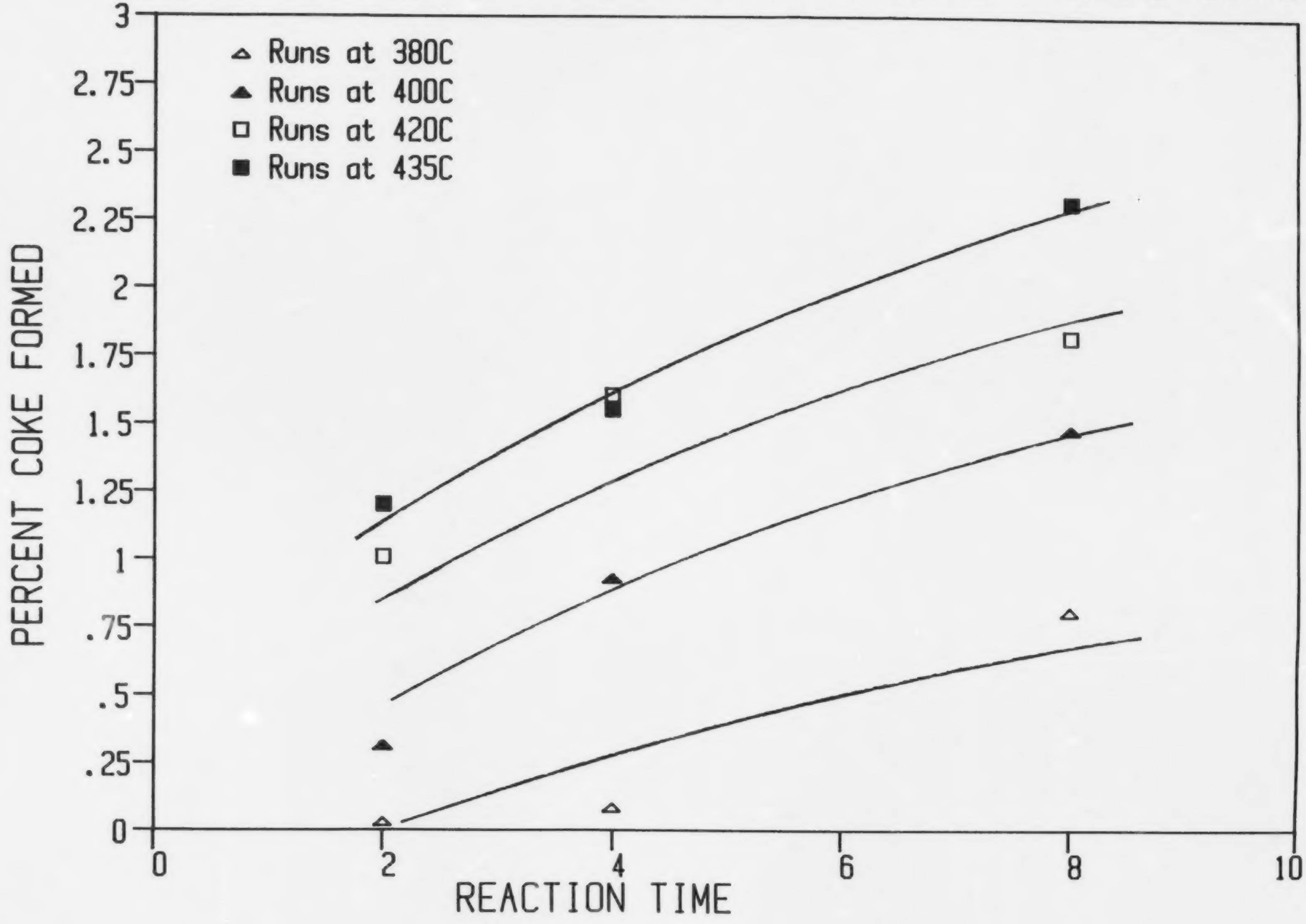

FIGURE 12: ARRHENIUS PLOTS FOR COKE FORMATION

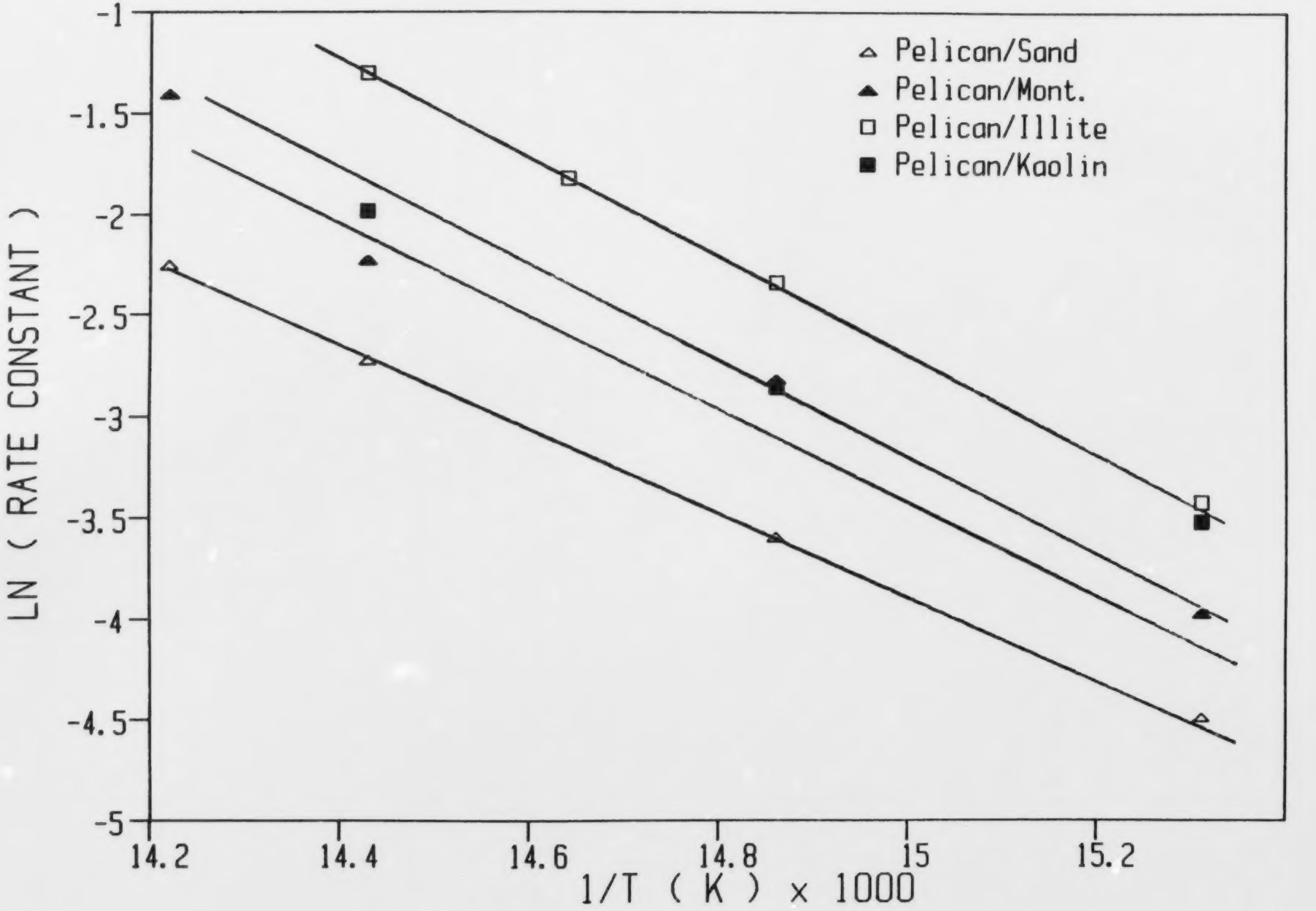

FIGURE 13: DELMAS/ILLITE - COKE ARRHENIUS PLOT

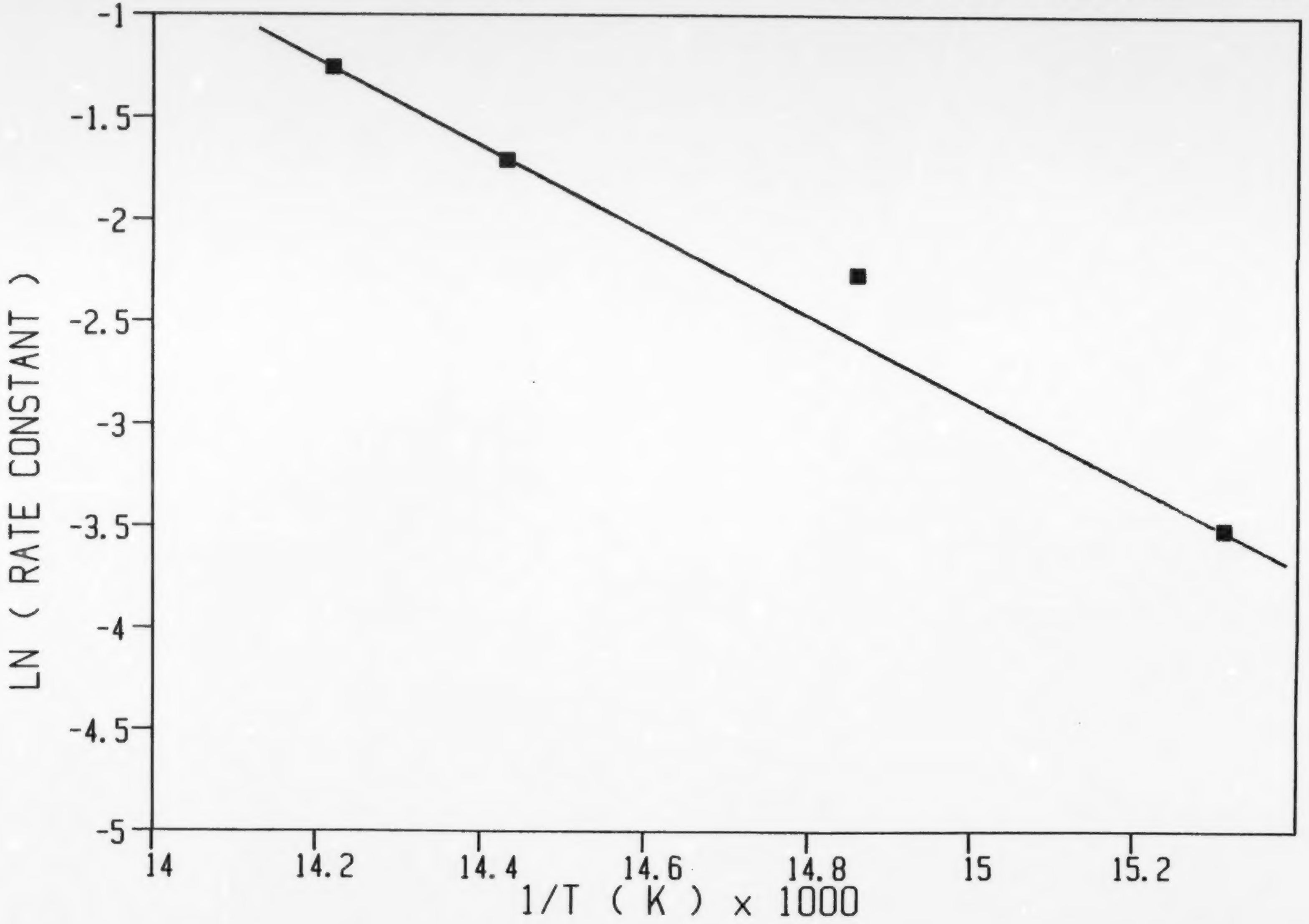

FIGURE 14

VISICALC TEMPLATE FOR INTERPOLATION

OF TGA TEMPERATURE AND dV/dt DATA

```
PELICAN RESID ON 90% MONTMOR. AT 5C/MIN     TGA RUN JULY 11          1985
INITIAL=              100.044
FINAL WT%=             93.431              FILE NAME      PELMON1
Vo=                     6.613              DATA DISC      TGA DATADISC III
REACTION  %    FRACTION       TGA WEIGHT %  DERIVATIVE     LN((1/Vo)*dV/dT
             10            .1         99.3827  .096407407407  -4.2282096488
             20            .2         98.7214            .14   -3.855150265
             30            .3         98.0601  .177215277777  -3.6194274355
             40            .4         97.3988   .19529281768  -3.5222926261
             50            .5         96.7375  .230818584071  -3.3551606362
             60            .6         96.0762  .261541984733  -3.2301978637
             70            .7         95.4149  .278916030535  -3.1658019172
             80            .8         94.7536  .234800000001  -3.3380585998
             90            .9         94.0923  .184790540541  -3.5775697171
--------------------------------------------------------------------------
SLOPE INTERPOLATION          PELICAN RESID/MONTMORILLONITE AT 5C/MIN
-----------------------------------------------------------------------------------------------
XONE          XTWO              XTHREE         YONE             YTWO          SLOPE          YTHREE
        99.427        99.346        99.3827              .08            .11  -.37037037037  .096407407407
        98.759        98.611        98.7214              .14            .14              0            .14
        98.164         98.02        98.0601              .17            .18 -.069444444444  .177215277777
        97.484        97.303        97.3988               .2            .19  .055248618784   .19529281768
        96.882        96.656        96.7375              .25            .22  .132743362831  .230818584071
        96.187        95.925        96.0762              .27            .25  .076335877862  .261541984733
        95.422         95.16        95.4149              .28            .24  .152671755725  .278916030535
        94.912        94.687        94.7536              .27            .22  .222222222222  .234800000001
        94.118         93.97        94.0923              .19            .16  .202702702702  .184790540541


PELICAN RESID ON 90% MONTMOR. AT 5C/MIN     TGA RUN JULY 11          1985
INITIAL=              100.044
FINAL WT%=             93.431              FILE NAME      PELMON2
Vo=                     6.613              DATA DISC      TGA DATADISC III
REACTION  %    FRACTION       TGA WEIGHT %  DERIVATIVE     LN((1/Vo)*dV/dT
             10            .1         99.3827    292.9345679   3.7909118575
             20            .2         98.7214    321.44486486  3.8837886265
             30            .3         98.0601    343.60763888  3.9504630129
             40            .4         97.3988    362.40651933  4.0037291544
             50            .5         96.7375    378.26902654  4.0465682439
             60            .6         96.0762    392.15679389  4.0826243352
             70            .7         95.4149    405.33278625  4.1156710153
             80            .8         94.7536    418.61999999  4.1479261783
             90            .9         94.0923    435.96824324  4.1885319952
--------------------------------------------------------------------------
SLOPE INTERPOLATION          PELICAN RESID/MONTMORILLONITE AT 5C/MIN
-----------------------------------------------------------------------------------------------
XONE          XTWO              XTHREE         YONE             YTWO          SLOPE          YTHREE
        99.427        99.346        99.3827            290.2          295.2 -61.7283950617    292.9345679
        98.759        98.611        98.7214            320.2          325.1 -33.1081081081   321.44486486
        98.164         98.02        98.0601              340            345 -34.7222222222   343.60763888
        97.484        97.303        97.3988            360.1            365 -27.0718232044   362.40651933
        96.882        96.656        96.7375            375.2            380 -21.2389380531   378.26902654
        96.187        95.925        96.0762              390          395.1 -19.4656488549   392.15679389
        95.422         95.16        95.4149            405.2          410.1 -18.7022900763   405.33278625
        94.912        94.687        94.7536            415.1          420.1 -22.2222222222   418.61999999
        94.118         93.97        94.0923            435.1          440.1 -33.7837837837   435.96824324
```

FIGURE 15

VISICALC TEMPLATE FOR SUMMARY OF TGA DATA

```
PELICAN RESID/ 90%ILLITE     SUMMARY OF DATA
FOR PLOTTING  BY METHOD OF   SHIH & SOHN   FRIEDMAN'S
                                           METHOD "
------------------------------------------------------------------------
FILE NAME :   PELIL9
DISK:         LUBES 2

------------------------------------------------------------------------
REACTION %                  10

TEMP RATE C/MINLN(I/Vo*dV/dT  TEMPERATURE C               I/T K
              5       -4.9578       308.737          .0017185171946
             10       -4.1941        323.66          .0016755470661
             15       -3.7936       332.865          .0016500969432
             20       -3.6316       339.883          .0016312069463
------------------------------------------------------------------------
REACTION %                  20
TEMP RATE C/MINLN(I/Vo*dV/dT  TEMPERATURE C               I/T K
              5       -4.4878       337.727          .0016369639557
             10       -3.8194       353.357          .0015961258833
             15       -3.4298        364.25          .0015688489355
             20       -3.1575       370.904          .0015526407314
------------------------------------------------------------------------
REACTION%                   30
TEMP RATE C/MINLN(I/Vo*dV/dT  TEMPERATURE C               I/T K-
              5       -4.2646       360.645          .0015777723432
             10       -3.5962       376.263          .0015398284323
             15        -3.234       387.439          .0015137776473
             20       -2.9252        394.61          .0014975216018
------------------------------------------------------------------------
REACTION %                  40

TEMP RATE C/MINLN(I/Vo*dV/dT  TEMPERATURE C               I/T K
              5       -4.0995         379.1          .0015331309600
             10        -3.387       394.552          .0014976516822
             15         -2.98       405.884          .0014726586201
             20        -2.677       412.617          .0014581999688
------------------------------------------------------------------------
REACTION %                  50
```

FIGURE 15 (Cont)

```
---------------------------------------------------------------------------
REACTION %                 50
TEMP RATE C/MINLN(I/Vo*dV/dT  TEMPERATURE C                  I/T K
               5        -3.8592          393.995             .0014989020543
              10        -3.1783              409             .0014659317462
              15         -2.772          420.448             .0014417365428
              20         -2.478          427.139             .0014279614850
---------------------------------------------------------------------------
REACTION %                 60
TEMP RATE C/MINLN(I/Vo*dV/dT  TEMPERATURE C                  I/T K
               5         -3.734          406.476             .0014713758541
              10        -3.0542          421.233             .0014401066831
              15         -2.624          432.473             .0014171672810
              20        -2.2978          438.878             .0014044194271
---------------------------------------------------------------------------
REACTION %                 70
TEMP RATE C/MINLN(I/Vo*dV/dT  TEMPERATURE C                  I/T K
               5         -3.734          417.716             .0014474377457
              10        -3.0084          432.053             .0014180112959
              15         -2.566          443.115             .0013961118286
              20        -2.2563          449.418             .0013839336376
---------------------------------------------------------------------------
REACTION %                 80
TEMP RATE C/MINLN(I/Vo*dV/dT  TEMPERATURE C                  I/T K
               5        -3.7636          428.762             .0014246597200
              10        -3.0523          442.871             .0013965875779
              15        -2.6255          453.877             .0013754458164
              20        -2.3329          459.945             .0013640610827
---------------------------------------------------------------------------
REACTION %                 90
TEMP RATE C/MINLN(I/Vo*dV/dT  TEMPERATURE C                  I/T K
               5         -4.205          442.409             .0013974892708
              10        -3.4377          455.687             .0013720300694
              15         -2.977          466.355             .0013522376152
              20        -2.6243          472.515             .0013410668187
```

FIGURE 16: TGA COKE LEVELS vs. CLAY CONTENT

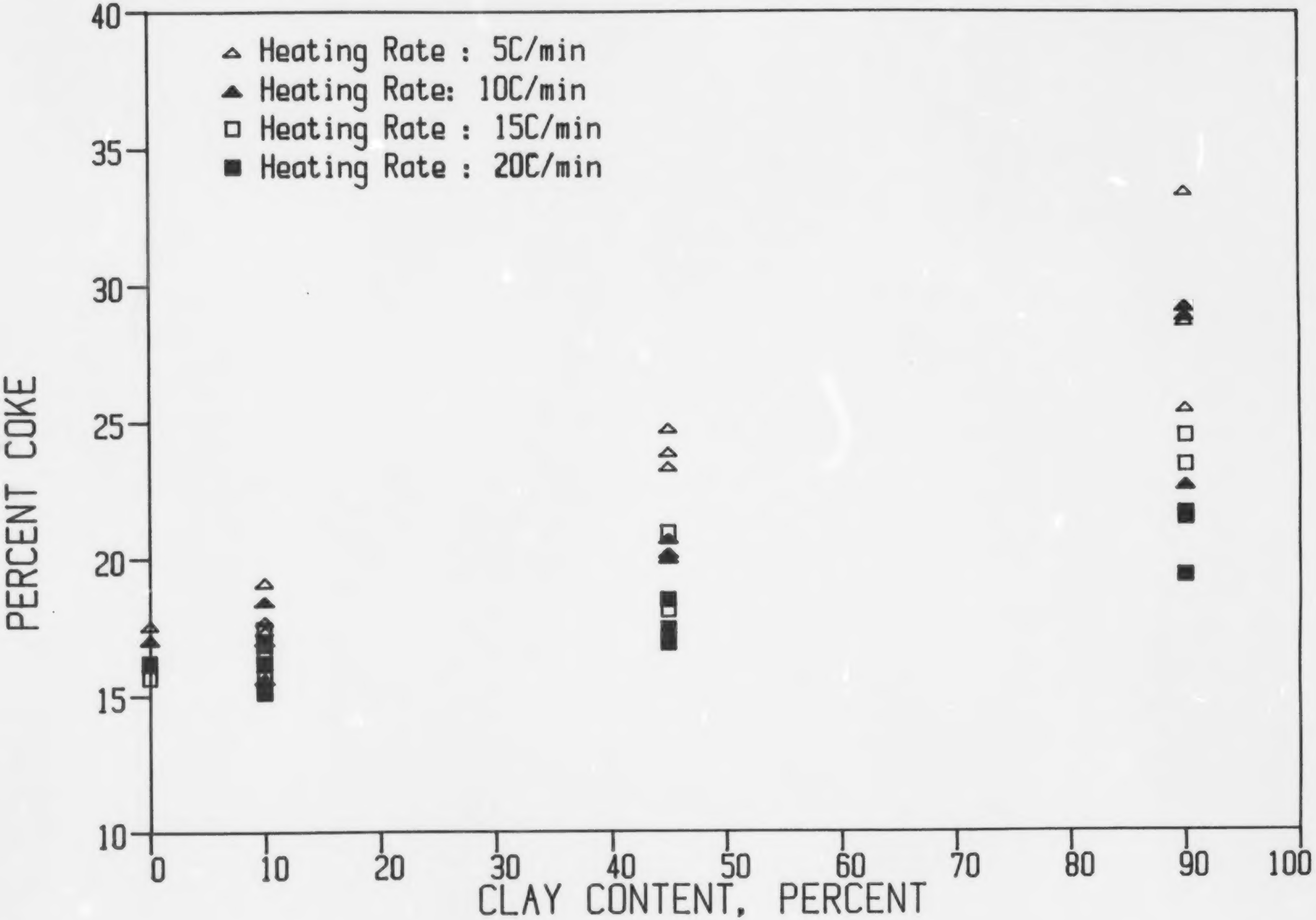

FIGURE 17: PELICAN RESID - Ea'S BY FRIEDMAN

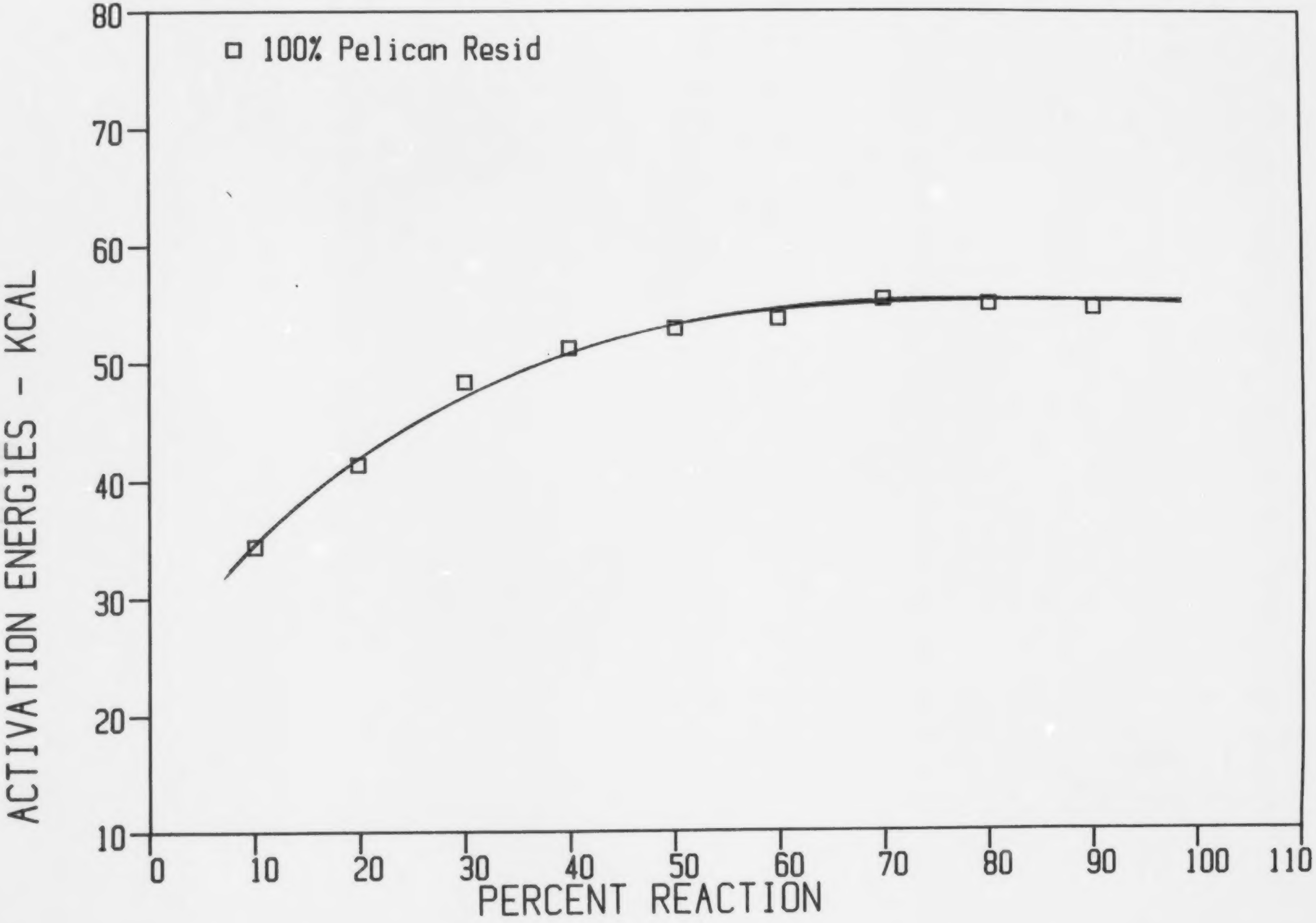

FIGURE 18: PELICAN/KAOLIN SAMPLES - FRIEDMAN

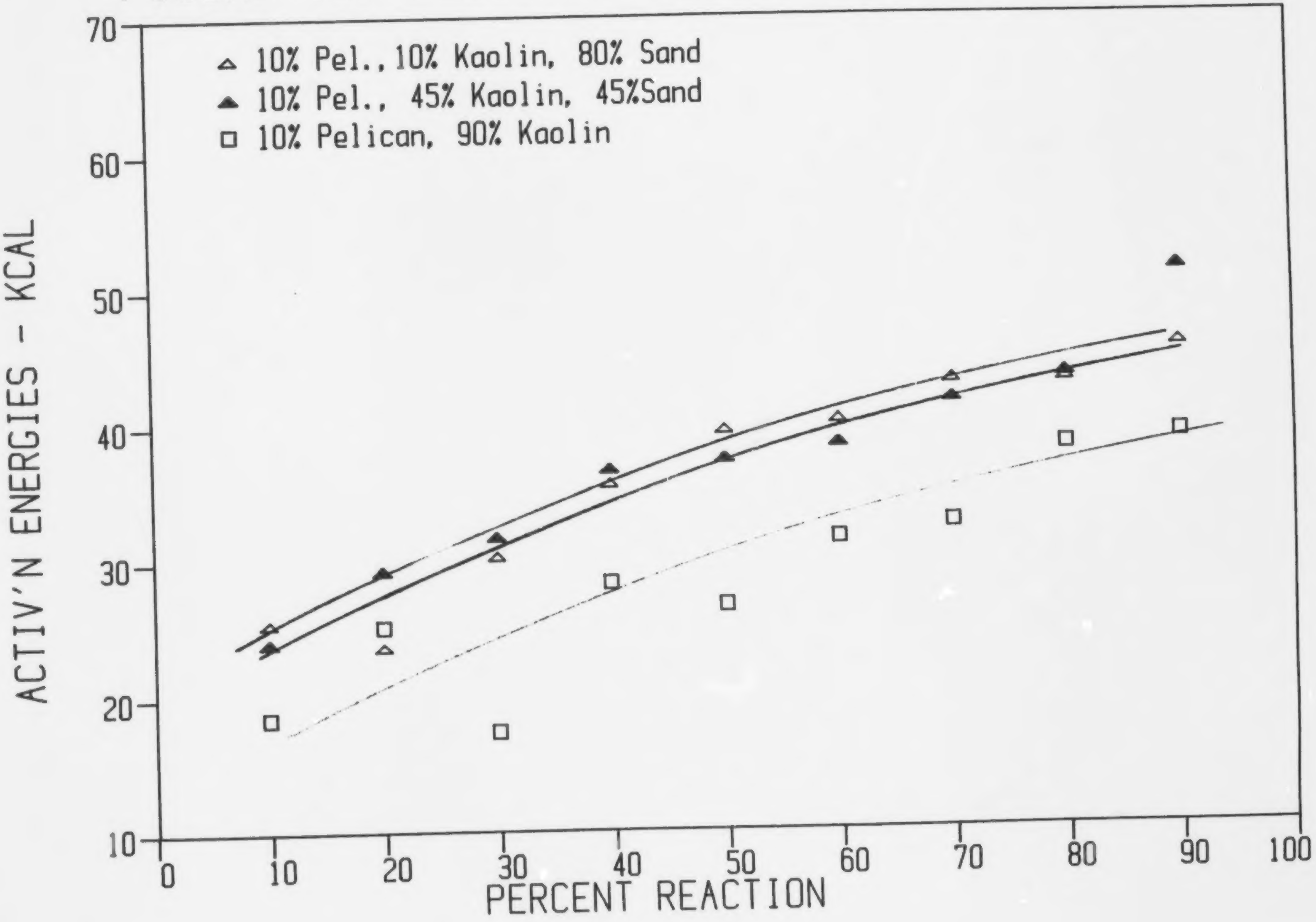

FIGURE 19: PELICAN/MONTMOR. SAMPLES-FRIEDMAN

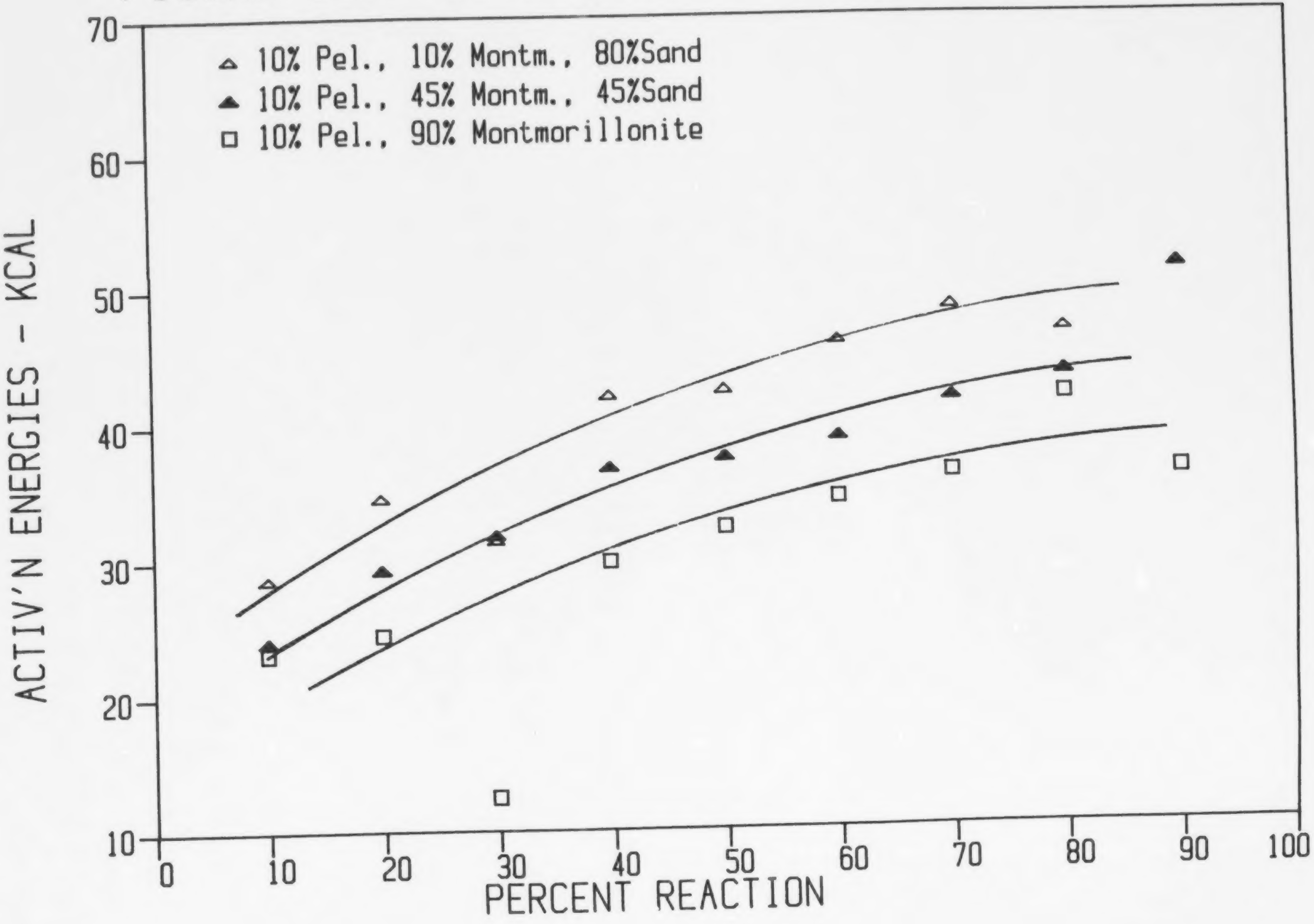

FIGURE 20: PELICAN/ILLITE SAMPLES - FRIEDMAN

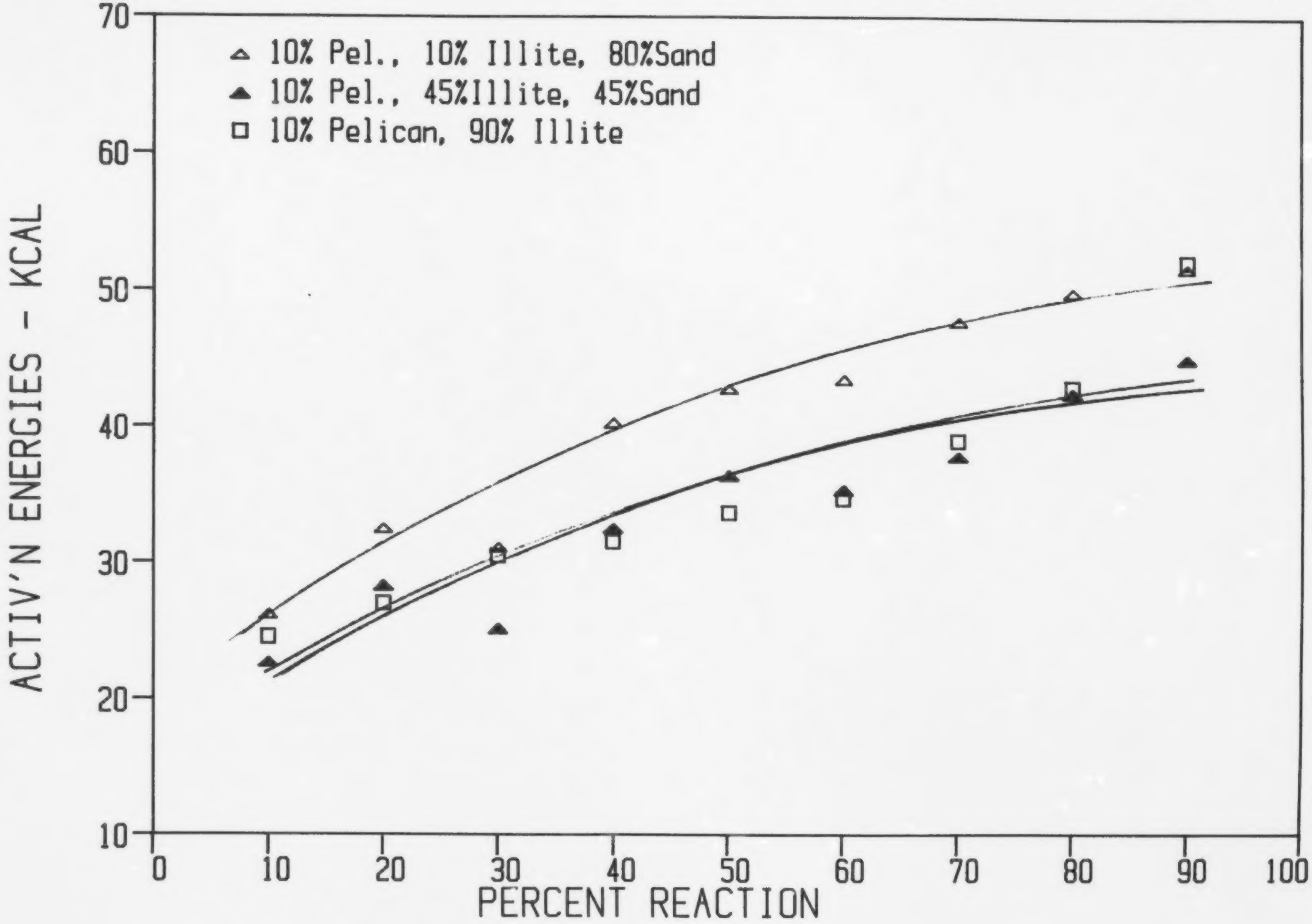

FIGURE 21: PELICAN AND PELICAN/SAND -FRIEDMAN

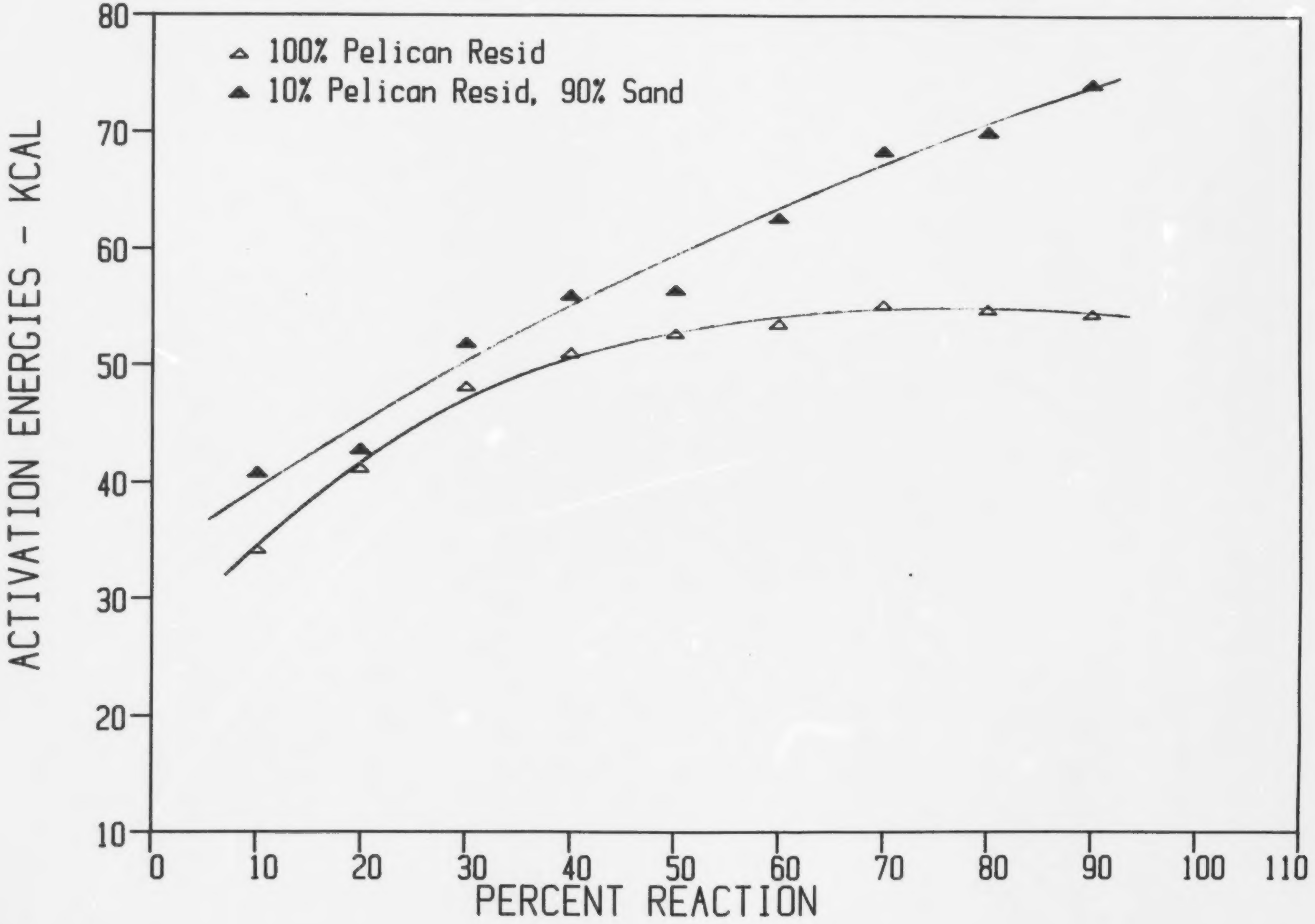

FIGURE 22: SMOOTHED Ea'S: PELICAN/KAOLIN

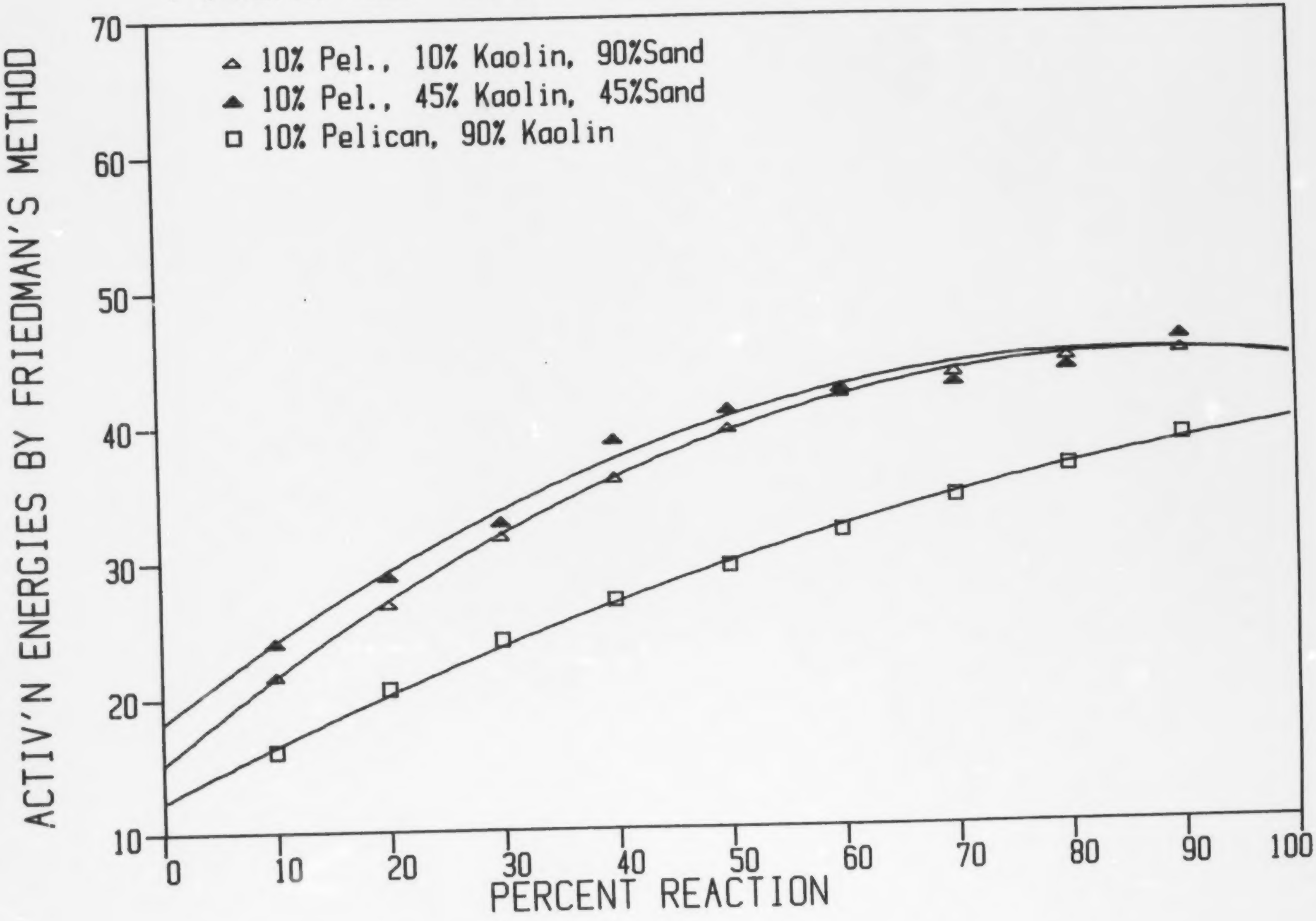

FIGURE 23: SMOOTHED Ea'S: PELICAN/MONTMORILLONITE

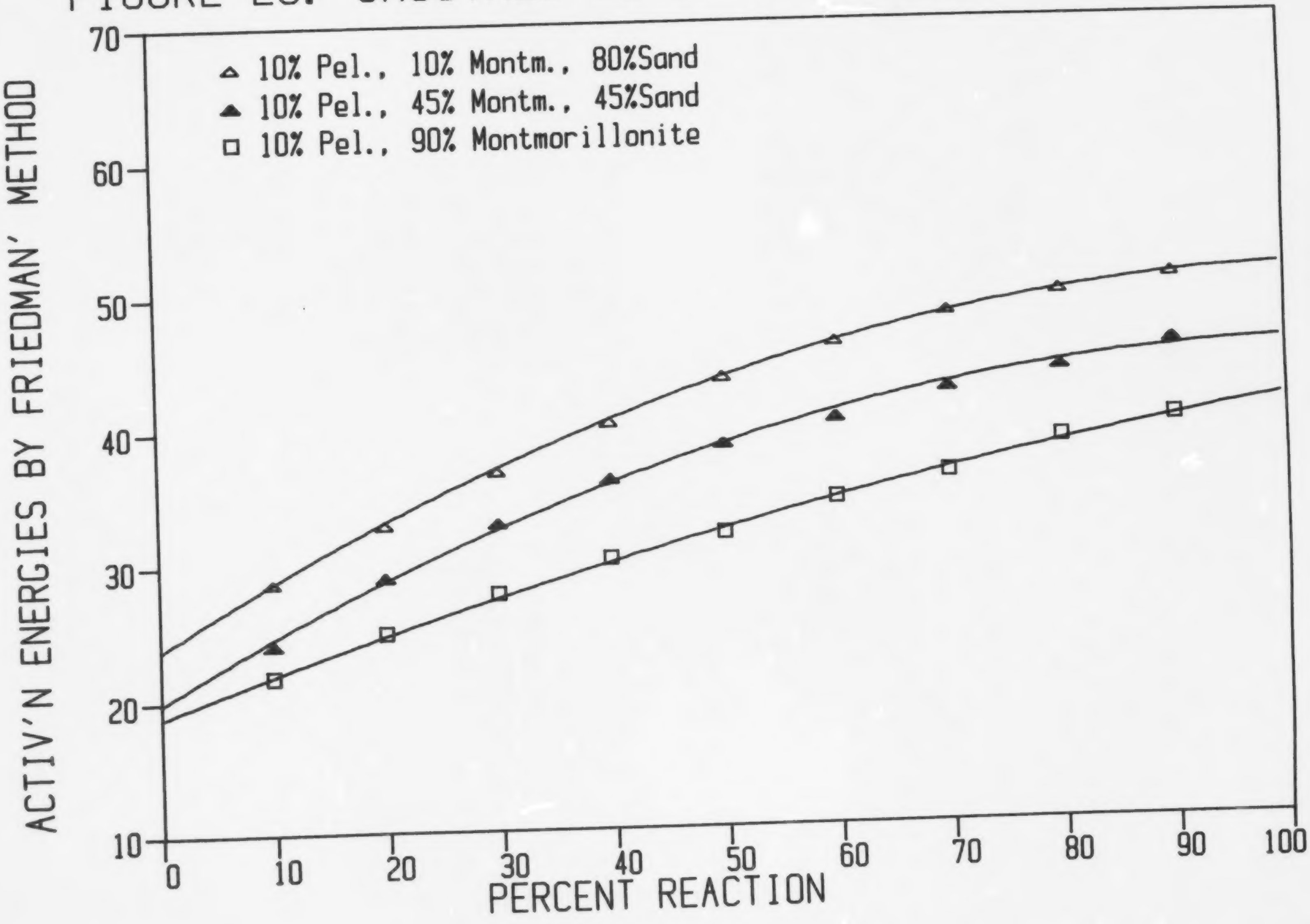

FIGURE 24: SMOOTHED Ea'S: PELICAN/ILLITE

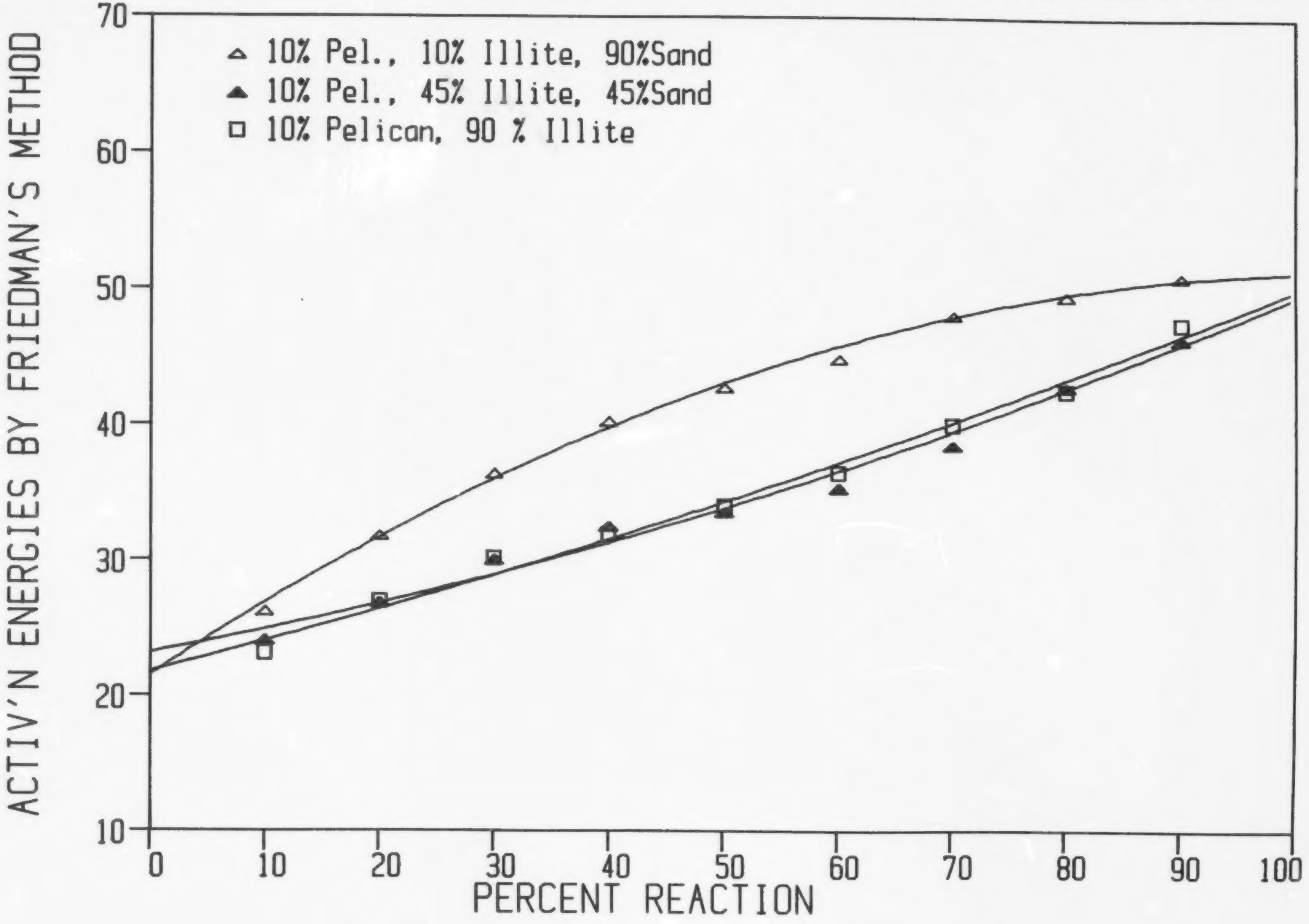

FIGURE 25: PELICAN/KAOLIN/SAND: ACTIV'N ENERGIES

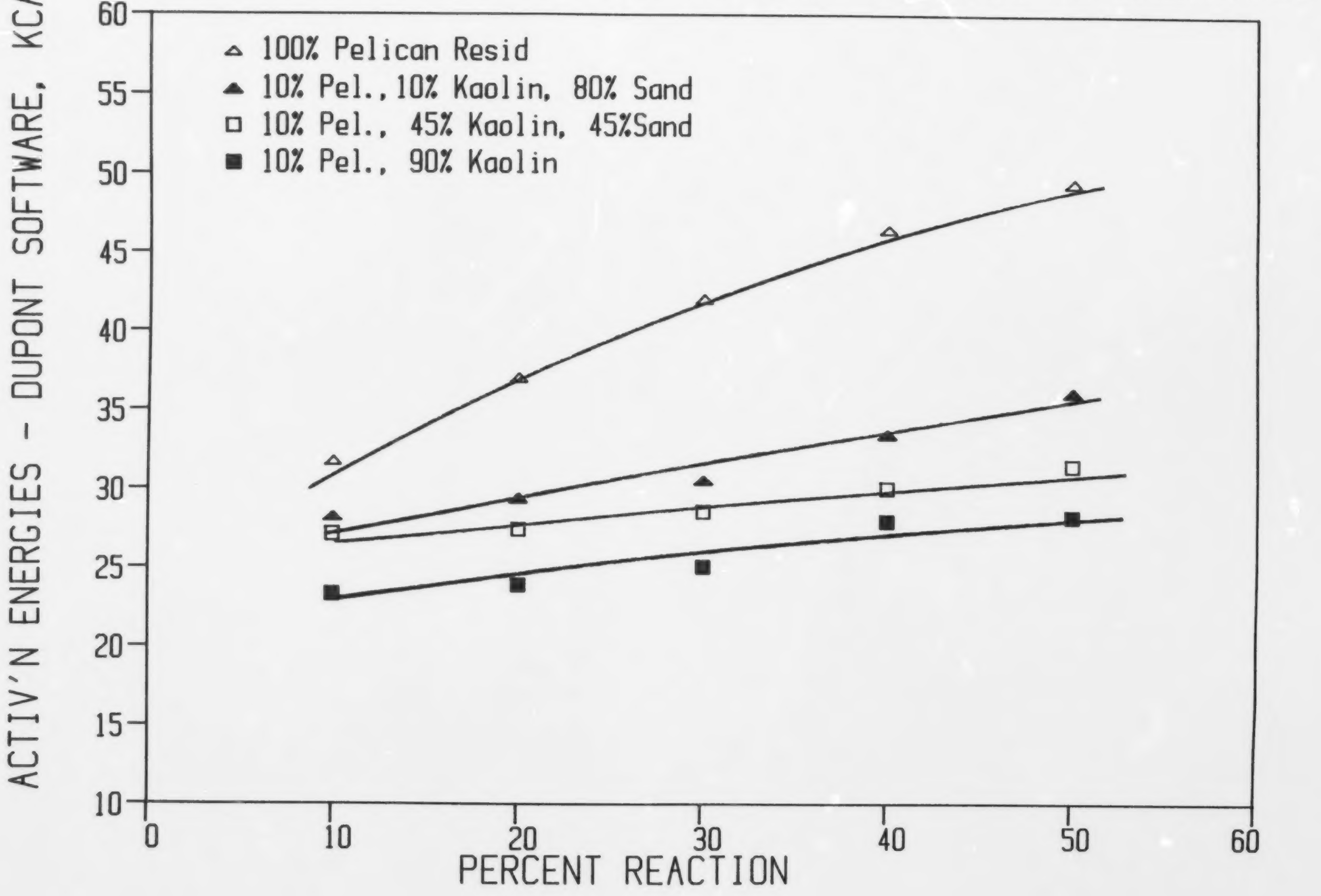

FIGURE 26: PELICAN/MONTM./SAND: ACTIV'N ENERGIES

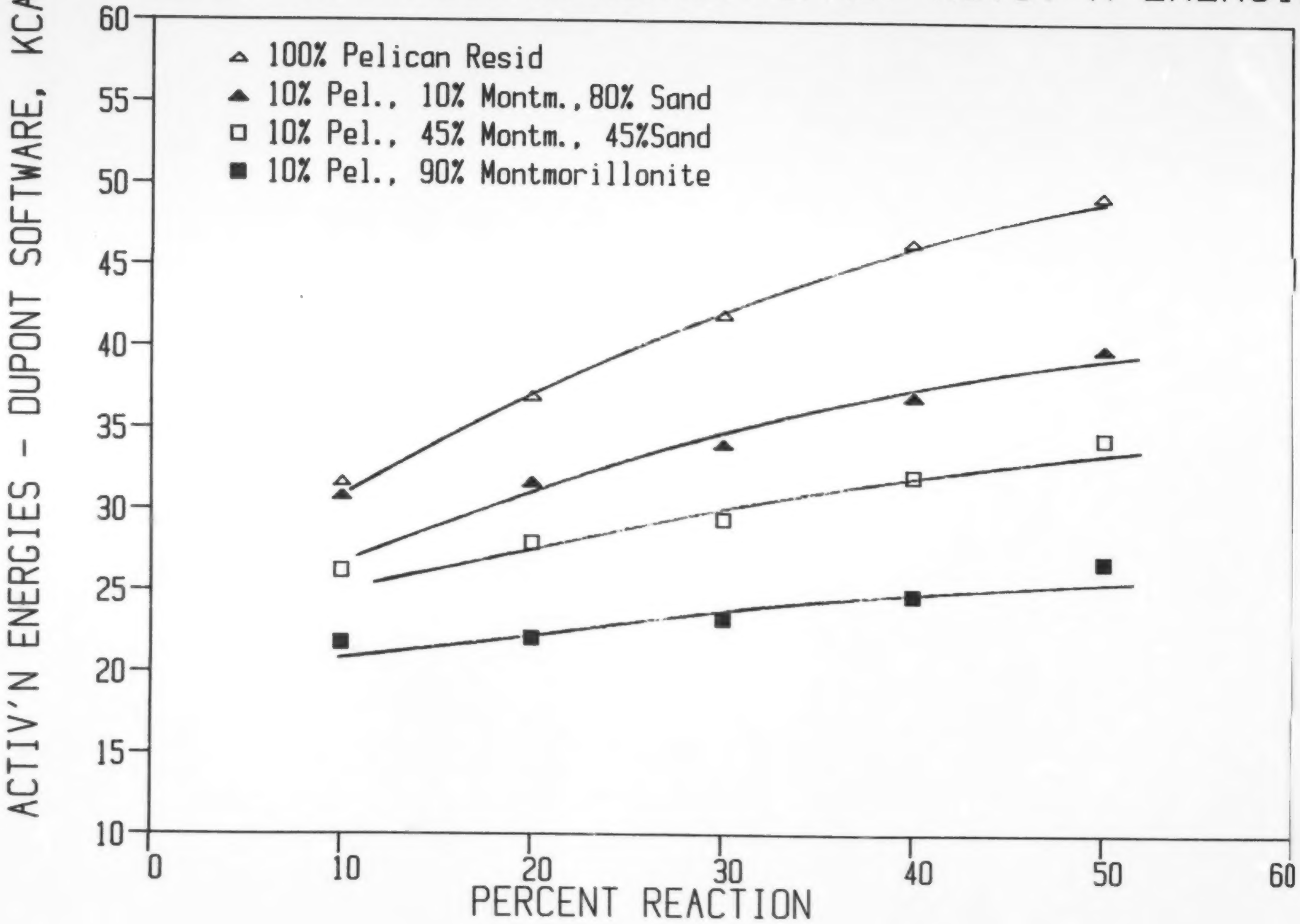

FIGURE 27: PELICAN/ILLITE/SAND: ACTIV'N ENERGIES

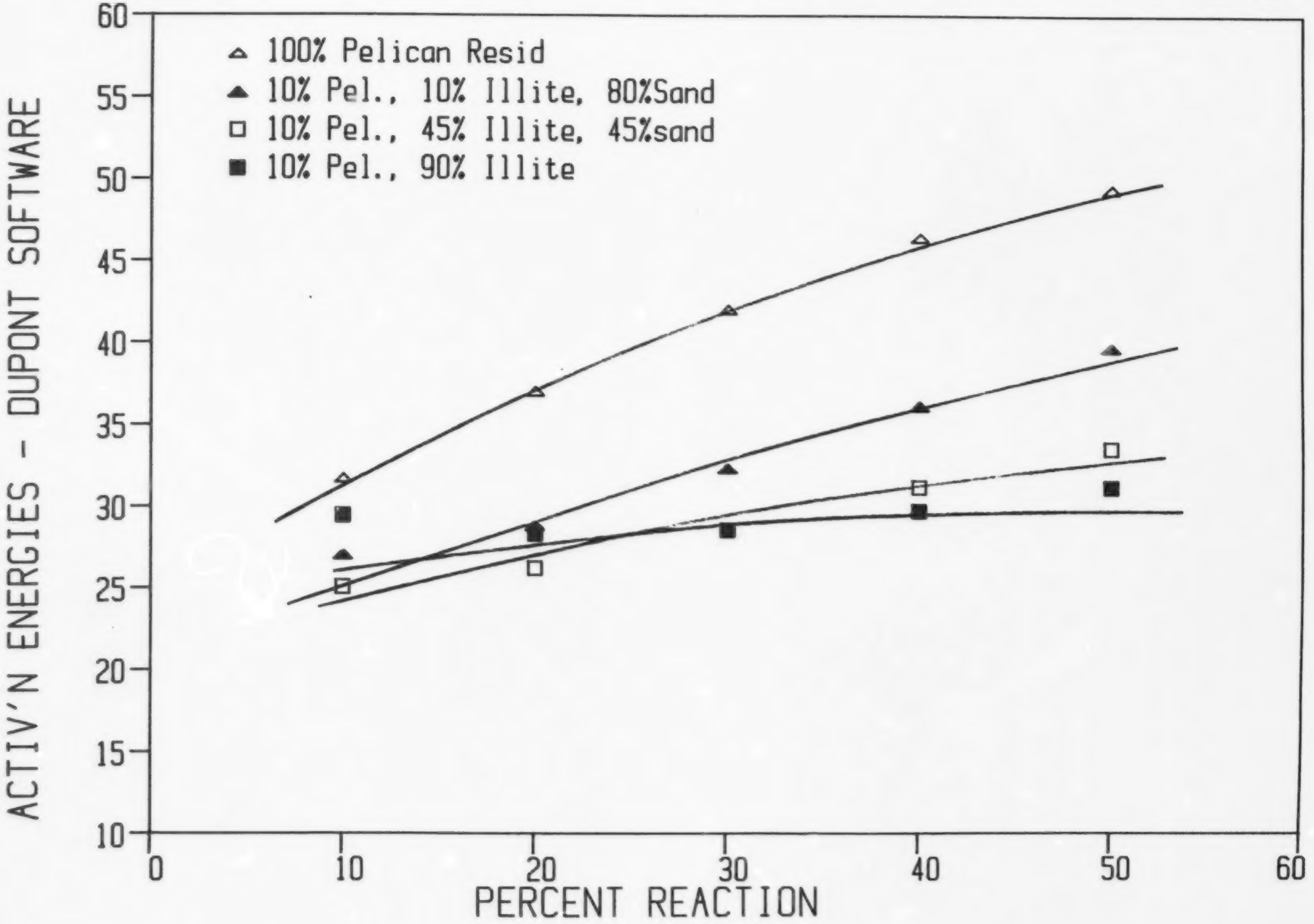

FIGURE 28: ARAB HEAVY RESID: ACTIV'N ENERGIES

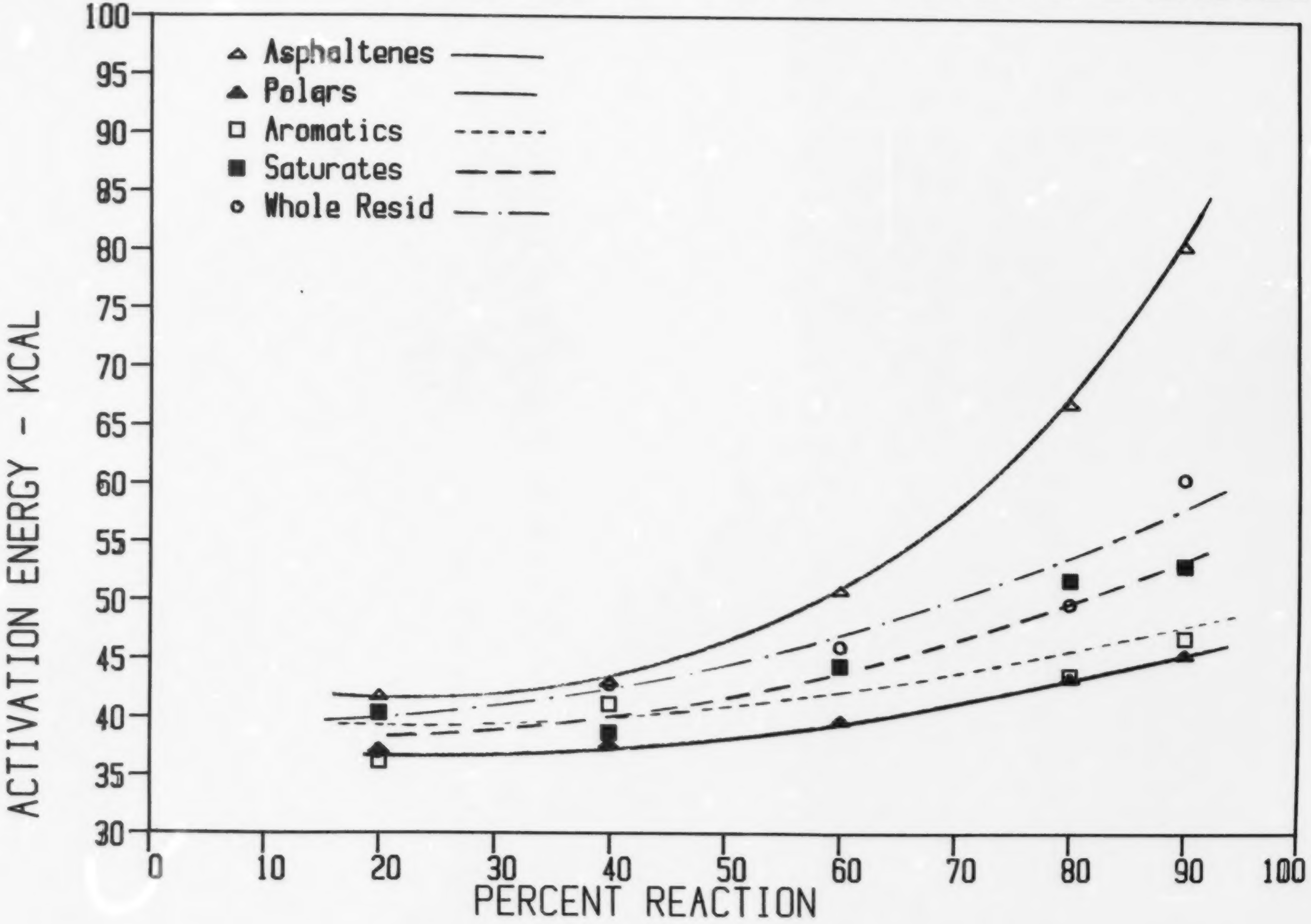

TABLE 1

KINETIC DATA FOR FUEL LAYDOWN USED IN NUMERICAL SIMULATIONS

Author	Reference	Cracking Kinetic Data		Comments
		A	EA	
Crookston et al	3	$3x10^5$	16	Taken from publications by Bousaid (17), Dabbous (25) and Burger (26) - these parameters are all for low temperature oxidations.
Culham	4	$1x10^7$	15	'Catalytic cracking' parameters for 26.5° API oil - source not given.
		$3.3x10^{20}$	62	Non-catalytic parameters for a 26.5° API oil - source not given.
		$4x10^6$	15	Catalytic parameters for 8.5° oil - source not given.
		$1x10^{16}$	47.5	Non-catalytic parameters for 8.5° API oil - source not given.
Bennion	1	$1x10^6$	45	Coke formation measured directly in autoclave experiments, but these were closed units with no sand present.
Bennion	2	$3.2x10^{12}$	17	Numbers used to make ISCOM work - arrived at by trial and error.

TABLE 1 (CONT'D)

Author	Reference	Cracking Kinetic Data		Comments
		A	EA	
Henderson	5	$8x10^7$	49	Visbreaking data, measured by % conversion; no measurement of coke.
McNab	6	-	-	Performed in relation to petroleum evolution, measurements by % conversion - no coke measurements.
Anis	7	$0.3x10^6$	40	Data 'chosen' for simulation.
Buchanan	8	Not given	18.5	ISCOM simulation of Lloydminster combustion tube - no source given. Others used appear to be 'standard literature values'.
Madgewick	9	Not given	16.5	Pyrolyses of Lloydminster oil in a fluidized bed at 500-800°C. Reaction times were extremely short, 1-8 seconds.
Thomas	10	-	14.04	Obtained from analyses of combustion tube runs - final data dependent on initial guesses.
Tadema & Weijdema	11	Variable - Crude Dependent	17-20	These are activation energies for LTO in the temperature range 60-120°C.

TABLE 1 (CONT'D)

Author	Reference	Cracking Kinetic Data		Comments
		A	EA	
N. Edmunds	12	$3x10^5$	16	Taken from Crookston.
L. Freeman	13	$1.8x10^7$	18.5	Adjusted value of Edmunds.
A. Chow	14	$12.4x10^7$	18.5	Adjusted value of Freeman.
Schucker	15	-	37-60	From non-isothermal TGA on residua; EA dependent on extent of reaction; performed to determine kinetics of coking (for Flexicoking) by weight loss.
Barbour	16	-	33-35	A Utah tar sand; measured by weight loss of a sample placed in a tube furnace boat.

Units, EA - kcal; A - $(\text{day})^{-1}$

TABLE 2

PROPERTIES OF PELICAN CRUDE OIL

Density		0.9694
API Gravity		14.4
Sulfur		5.16
Nitrogen, %		0.29
Nickel, ppm		44.3
Vanadium, ppm		139
Ramsbottom Carbon, %		8.76
Viscosity, cSt	@ 25C	1038
	@ 40C	321
C5 Insolubles, %		15.0
Distillation, TBP, C		
	IBP	71
	10 %	220
	30 %	360
	50 %	
	FBP	504

TABLE 3

PROPERTIES OF DELMAS CRUDE OIL

Density	1.008
API Gravity	9.8
Sulfur, mass %	4.3
Nitrogen, mass %	0.39
Nickel, ppm	60
Vanadium, ppm	160
Ramsbottom Carbon	10.0
C5 Insolubles	18.0
Distillation, GCD, C	
IBP	106
5 %	198
10%	244
30%	354
50%	445
70%	540
90%	637

TABLE 4

COKE LEVELS AS MEASURED BY CARBON CONTENT ON COMPOSITES PRODUCED IN ISOTHERMAL REACTOR RUNS WITH PELICAN/SAND.

Run #	Temp. C	Time, h	% Coke	Ave. % Coke
21	380	2	0.01	0.01
18	380	4	0.05, 0.014, 0.015, 0.023	0.03
23	380	8	0.18, 0.17, 0.206,0.213	0.19
10	400	2	0.09, 0.10	0.09
12	400	4	0.26, 0.209, 0.202	0.26
14	400	4	0.29	0.29
15	400	8	0.53, 0.34, 0.35, 0.31, 0.37	0.38
57	420	2	0.34, 0.37, 0.87*, 0.76*	0.36
17	420	4	0.54, 0.46, 0.54, 0.52	0.54
34	420	8	0.54, 0.65, 0.60, 1.0, 0.62	0.68
32	435	2	0.45, 0.46	0.46
30	435	4	0.69, 0.69	0.69
35	435	8	0.91, 0.99, 1.22, 1.43*, 0.88, 0.86, 1.36	1.04

* Denotes value omitted from averaged coke calculation.

TABLE 5

COKE LEVELS AS MEASURED BY CARBON CONTENT ON COMPOSITES PRODUCED IN ISOTHERMAL REACTOR RUNS WITH PELICAN/KAOLIN/SAND.

Run #	Temp. C	Time, h	% Coke	Ave. % Coke
24	380	2	0.238, 0.215	0.23
19	380	4	0.35, 0.3	0.33
27	380	8	0.398, 0.394, 0.5	0.43
13	400	2	0.27	0.27
78	400	3.5	0.38, 0.40, 0.48, 0.41	0.42
16	400	8	0.65	0.65
26	420	2	0.571, 0.582, 0.641, 0.635	0.58
20	420	4	0.8, 0.847, 0.883, 0.843	0.84
53	420	4	0.85, 0.74, 0.92, 0.68, 0.76	0.79
66	420	8	0.86, 1.21, 0.93, 0.99	1.0
28	435	2	0.749, 0.752, 0.832, 0.81, 0.89, 0.82, 0.85, 0.83	0.78
56	435	2	0.75, 0.71, 0.68, 0.71, 0.81 0.89, 0.82, 0.85, 0.83	0.78
29	435	4	0.675, 0.683, 0.69, 0.70, 0.73, 0.75, 0.76	0.71
33	435	8	0.54, 0.72, 0.80, 0.79, 0.90	0.75
68	435	8	0.72, 0.88, 0.81, 0.81	0.81

TABLE 6

COKE LEVELS AS MEASURED BY CARBON CONTENTS ON COMPOSITES PRODUCED IN ISOTHERMAL RUNS WITH PELICAN/MONTMORILLONITE/SAND

Run #	Temp. C	Time, h	% Coke	Ave % Coke
36	380	2	0.022, 0.03, 0.03, 0.045, 0.056	0.037
38	380	4	0.06, 0.051, 0.062, 0.075 0.079	0.065
65	380	4	0.03, 0.05, 0.07, 0.08	0.06
42	380	8	0.281, 0.286, 0.326, 0.363 0.443*	0.31
37	400	2	0.094, 0.01*,0.164, 0.119, 0.21	0.15
39	400	4	0.356, 0.436, 0.735*, 0.51, 0.578, 0.617, 0.763*	0.50
44	400	8	0.449*, 0.423*, 0.49*, 0.657 1.13, 0.856, 0.723	0.84
41	420	2	0.517, 0.72*, 0.542, 0.79*	0.59
40	420	4	0.741, 0.689, 0.694, 0.724	0.71
43	420	8	1.124, 1.035, 0.713*, 0.853	1.00
58	435	2	0.831, 0.696, 0.737, 0.646	0.73
59	435	4	1.044, 1.254, 0.661*, 1.349	1.22
63	435	8	1.74, 1.87, 2.09*, 1.58	1.73

*** Denotes value omitted from averaged coke calculation.**

TABLE 7

COKE LEVELS AS MEASURED BY CARBON CONTENTS ON COMPOSITES PRODUCED FROM ISOTHERMAL RUNS WITH PELICAN/ILLITE/SAND.

Run #	Temp. C	Time, h	% Coke	Ave % Coke
45	380	2	0.018, 0.014	0.016
46	380	4	0.047, 0.043	0.045
64	380	4	0.04, 0.06, 0.07, 0.05	0.055
49	380	8	0.341, 0.498, 0.675*, 0.487 0.596	0.48
48	400	2	0.234, 0.404, 0.635, 0.277, 0.465	0.40
47	400	4	0.48, 0.71, 0.39*, 0.84*, 0.50	0.56
54	400	8	1.18, 1.43, 1.37, 1.16	1.28
62	410	2	0.43, 0.71, 0.74, 0.54	0.60
61	410	4	0.81, 0.98, 1.11, 1.39.33,	1.07
60	410	8	0.955, 1.322, 4.03*, 1.22	1.17
51	420	2	0.71, 0.91, 1.05, 0.96, 0.67	0.86
50	420	4	0.867, 1.184, 1.316,1.33, 1.36, 0.93, 0.95	1.13
52	420	8	1.04*, 1.65, 1.98, 1.36, 1.5 1.08*	1.63

* Denotes value omitted from averaged coke calculation.

TABLE 8

COKE LEVELS AS MEASURED BY CARBON CONTENTS ON COMPOSITES PRODUCED FROM ISOTHERMAL RUNS WITH DELMAS/ILLITE/SAND.

Run #	Temp. C	Time, h	% Coke	Ave % Coke
70	380	2	0.033, 0.064, 0.022, 0.055	0.04
69	380	4	0.03*, 0.09, 0.12, 0.05	0.09
71	380	8	0.563, 0.725, 0.905, 1.052*	0.73
74	400	2	0.222, 0.29, 0.387, 0.37	0.32
73	400	4	0.543, 0.659, 0.99, 1.571	0.94
72	400	8	1.007, 1.532, 1.332, 2.037	1.48
75	420	2	0.716, 0.857, 0.502, 1.494, 1.43	1.0
80	420	4	1.363, 1.933, 1.535, 2.469*, 2.284*	1.61
76	420	8	1.313*,1.749, 2.387	1.82
77	435	2.5	1.242, 1.319, 1.128, 1.122	1.2
78	435	4	0.575*, 0.817*, 1.548, 0.639*	1.55
79	435	8	2.626, 1.758*, 2.15, 2.187	2.32

* Denotes value omitted from averaged coke calculation

TABLE 9

ISOTHERMAL REACTOR PRODUCTS, MASSES OF MATERIAL FROM TOP, MIDDLE, AND BOTTOM OF REACTOR, AND COKE CONTENTS.

Run #	% Carbon			Mass, g		
	Top	Middle	Bottom	Top	Middle	Bottom
1	0.5	0.51	2.68	na	na	na
4	0.36	0.12	0.69	na	na	na
5	0.28	0.31	0.43	na	na	na
6	0.24	0.26	0.22	na	na	na
7	0.30	0.33	0.51	na	na	na
8	0.32	0.37	0.50	na	na	na
9	6.48	2.79	1.49	na	na	na
10	0.01 0.01	0.03 0.02	0.14 0.14	40.5	94.2	46.84
11	0.02	0.02	0.03	46.2	89.7	46.3
12	0.01 0.02	0.06 0.04	0.61 0.83	41.6	98.4	40.9
13	0.07	0.33	0.39	42.3	79.5	67.5
14	0.17	0.48	0.29	44.7	na	41.62
15	0.06 0.01	0.23 0.18	0.65 0.70	43.12	93.82	41.25
16	0.58	0.50	0.71	40.28	98.48	40.11
17	0.10 0.09	0.36 0.33	1.04 3.8	40.43	96.76	40.19
18	0.05	0.05	0.06	38.94	93.3	47.96
19	0.24	0.33	0.42	52.92	87.00	40.39
20	0.73	0.56	0.83	41.06	96.23	42.22
21	0.01	0.03	0.03	43.00	96.4	44.45
22	0.01	0.10	1.09	45.00	91.30	44.00

TABLE 9 (cont.)

Run #	% Coke			Mass, g		
	Top	Middle	Bottom	Top	Middle	Bottom
23	0.01	0.02	0.19	40.10	89.80	41.23
24	0.16 0.16	0.22 0.21	0.24 0.25	43.80	97.76	43.04
26	0.36 0.39	0.60 0.58	0.62 0.64	41.91	77.82	41.50
27	0.30	0.39	0.40	41.50	84.27	41.55
28	0.78	0.79	0.98	40.55	85.84	43.60
29	0.63	0.65	0.74	43.35	86.44	47.29
30	0.44	0.33	1.74	42.93	93.11	42.54
31	0.85	0.74	0.69	43.07	87.11	44.00
32	0.09	0.22	3.41	44.40	90.92	43.70
33	0.67	0.83	0.70	43.00	85.22	43.3
34	0.83	0.36	1.40	42.82	90.26	42.31
35	0.57	0.51	2.96	43.07	89.50	43.78
36	0.02	0.02	0.03	37.08	74.7	37.76
37	0.02	0.07	0.20	36.6	81.80	37.43
38	0.02	0.03	0.05	38.4	70.73	37.87
39	0.03	0.53	0.50	36.64	75.70	36.76
40	1.02	0.67	0.77	38.24	76.13	37.76
41	0.37	0.71	0.75	36.22	79.96	38.15
42	0.03	0.21	0.58	38.71	80.10	39.77
43	1.38	0.64	0.58	38.34	76.82	36.07
44	0.34	0.81	0.72	37.63	74.14	39.02
45	0.01	0.01	0.03	37.68	80.84	34.80
46	0.04	0.04	0.10	37.18	73.27	37.73

TABLE 9 (Cont.).

Run #	Coke %			Mass, g		
	Top	Middle	Bottom	Top	Middle	Bottom
47	0.02	0.90	1.14	38.21	72.71	36.57
48	0.10	0.27	0.63	37.70	72.97	38.87
49	0.02	0.37	0.69	38.12	75.24	37.57
50	0.60	0.90	1.10	40.62	69.76	37.59
51	0.05	1.07	1.28	38.73	73.10	32.63
52	0.60	1.64	1.17	39.29	64.78	39.33
53	0.77	0.75	0.77	37.23	70.54	38.28
54	0.38	1.07	1.18	39.52	65.63	39.00
55	0.49	0.66	0.61	39.11	71.12	39.00
56	0.39	0.52	0.62	39.9	69.92	38.47
57	0.01	0.05	2.14	39.72	75.91	38.00
58	0.21	0.73	0.73	39.28	76.41	39.00
59	1.34	0.70	0.84	38.85	73.82	38.13
60	0.95	0.92	0.96	39.00	68.00	39.11
61	0.20	0.95	1.13	38.79	64.10	38.00
62	0.02	0.56	1.06	38.00	72.68	37.89
63	0.97	0.51	0.70	38.86	74.67	37.96
64	0.01	0.03	0.08	37.52	77.36	36.91
65	0.03	0.03	0.06	38.05	76.23	36.64
66	0.79	0.81	0.86	35.92	65.47	36.62
67	0.50	0.72	0.80	37.85	71.30	38.88
68	0.75	0.55	0.78	38.57	65.95	38.50
69	0.03	0.11	0.05	35.93	68.41	36.08
70	0.02	0.02	0.03	36.43	64.71	37.50

TABLE 9 (Cont.)

Run #	Coke %			Mass, g		
	Top	Middle	Bottom	Top	Middle	Bottom
71	0.18	0.53	0.76	35.67	63.10	36.33
72	0.03	1.32	1.17	36.71	65.21	35.07
73	0.02	0.86	0.93	37.77	70.78	36.42
74	0.02	0.14	0.20	36.60	73.16	36.42
75	0.88	2.04	1.46	37.23	73.02	37.07
76	0.04	1.34	1.60	37.06	75.77	38.22
77	0.74	0.99	1.50	38.04	66.90	38.08
78	0.57	0.86	0.64	37.00	61.30	37.00
79	3.27	1.51	2.02	37.00	61.59	37.00
80	0.60	1.37	3.05	37.00	75.44	37.11
81	0.72	0.70	0.92	37.98	73.68	38.47
82	0.15	1.21	1.75	38.53	68.82	38.13
83	0.69	0.62	0.73	38.94	75.68	39.40

TABLE 10: RATE CONSTANTS CALCULATED FOR COKE FORMATION IN ISOTHERMAL REACTOR RUNS.

Sample	Temperature	Time, h	k_{calc}	Coke$_{obs}$	Coke$_{calc}$
Pelican/Sand	380	2	0.005	0.02	0.036
	380	4	0.0128	0.10	0.07
	380	8	0.010	0.15	0.14
		Average =	0.011		
	400	2	0.026	0.10	0.11
	400	4	0.033	0.25	0.21
	400	8	0.024	0.35	0.40
		Average =	0.028		
	420	2	0.067	0.25	0.25
	420	4	0.072	0.50	0.46
	420	8	0.059	0.75	0.82
		Average =	0.066		
	435	2	0.111	0.40	0.38
	435	4	0.104	0.68	0.69
	435	8	0.100	1.10	1.14
		Average =	0.105		
Pelican/Mont.	380	2	0.005	0.02	0.06
	380	4	0.022	0.17	0.11
	380	8	0.017	0.25	0.22
		Average =	0.019		
	400	2	0.053	0.20	0.23
	400	4	0.069	0.48	0.43
	400	8	0.061	0.77	0.77
		Average =	0.061		
	420	2	0.127	0.45	0.39
	420	4	0.108	0.70	0.70
	420	8	0.087	1.00	1.15
		Average =	0.107		
	435	2	0.231	0.74	0.78
	435	4	0.245	1.25	1.25
	435	8	0.260	1.75	1.72

TABLE 10 (Cont.)

Sample	Temperature	Time, h	k_{calc}	$Coke_{obs}$	$Coke_{calc}$
Pelican/Kaolin	380	2	0.053	0.20	0.16
	380	4	0.041	0.30	0.30
	380	8	0.028	0.40	0.55
		Average =	0.040		
	400	2	0.068	0.26	0.22
	400	4	0.056	0.40	0.41
	400	8	0.049	0.65	0.74
		Average =	0.058		
	420	2	0.197	0.65	0.48
	420	4	0.124	0.78	0.84
	420	8	0.089	1.00	1.32
		Average =	0.136		
Pelican/Illite	380	2	0.006	0.02	0.08
	380	4	0.029	0.20	0.16
	380	8	0.036	0.45	0.31
		Average =	0.033		
	400	2	0.119	0.38	0.32
	400	4	0.091	0.55	0.58
	400	8	0.080	0.85	0.97
		Average =	0.097		
	410	2	0.203	0.60	0.50
	410	4	0.147	0.80	0.86
	410	8	0.137	1.20	1.31
		Average =	0.162		
	420	2	0.279	0.77	0.75
	420	4	0.236	1.10	1.19
	420	8	0.295	1.63	1.59
		Average =	0.270		

TABLE 10 (Cont.)

Sample	Temperature	Time, h	k_{calc}	$Coke_{obs}$	$Coke_{calc}$
Delmas/Illite	380	2	0.004	0.02	0.04
	380	4	0.010	0.10	0.07
	380	8	0.077	0.15	0.14
		Average =	0.009		
	400	2	0.099	0.45	0.47
	400	4	0.108	0.88	0.85
	400	8	0.103	1.40	1.41
		Average =	0.103		
	420	2	0.202	0.83	0.76
	420	4	0.179	1.28	1.28
	420	8	0.159	1.8	1.91
		Average =	0.180		
	435	2	0.319	1.18	1.08
	435	4	0.247	1.57	1.70
	435	8	0.288	1.25	2.24
		Average =	0.285		

TABLE 11

ACTIVATION ENERGIES FOR COKE FORMATION FROM ISOTHERMAL REACTOR DATA

Crude/Matrix Blend	Activation Energy, Kcal/mole
Pelican/Sand	40.5
Pelican/Sand/Mont.	43.6
Pelican/Sand/Illite	47.6
Pelican/Sand/Kaolin	~40
Delmas/Sand/Illite	37.8

TABLE 12

PROPERTIES OF PELICAN 504+C DISTILLATION RESIDUE

Density	1.0396
API Gravity	4.5
Sulfur, mass %	6.82
Nitrogen, %	0.52
Nickel, ppm	85.3
Vanadium, ppm	250
Ramsbottom Carbon, %	20.4
Viscosity, cSt @100C	3342
cSt @200C	45
Distillation, GCD, C	
% of Distilled	
IBP	185
5 %	413
10 %	489
20 %	528
30 %	550
40 %	567
50 %	581

TABLE 13

COKE FORMATION BY SAMPLE TYPE AS A FUNCTION OF HEATING RATE

Heating Rate C/Min	Mass % Coke 100% Pel.	10% Pel* 90% Sand	10% Pel* 80% Sand 10% Kaol	10% Pel 45% Sand 45% Kaol	10% Pel 90% Kaol
5	15.9	17.6	19.2	24.8	33.5
10	15.3	17.1	18.5	20.0	29.3
15	14.9	15.7	17.5	18.4	24.5
20	14.8	16.2	16.8	17.5	21.6
			10% Pel* 80% Sand 10% Mont	10% Pel 45% Sand 45% Mont	10% Pel 90% Mont
5			17.5	23.4	28.7
10			15.7	20.2	28.9
15			15.7	18.1	23.4
20			15.1	16.9	21.4
			10% Pel 80% Sand 10% Ill	10% Pel 45% Sand 45% Ill	10% Pel* 90% Ill
5			17.8	24.0	25.5
10			17.0	20.8	22.7
15			16.3	21.0	21.4
20			16.1	18.5	19.3

* Result is the average of two runs

TABLE 14

COKE LEVEL DETERMINATION BY TGA: DUPLICATE RESULTS

	% Coke Formed			
Heating Rate C/min	10% Pel. 90% Sand	10% Pel. 90% Sand	10% Pel. 10% Kaol 80% Sand	10% Pel. 10% Kaol 80% Sand
5	19.3	15.8	20.1	18.2
10	19.2	14.9	19.6	17.3
15	16.7	14.7	18.3	16.8
20	17.9	14.4	17.9	17.7

	10% Pel. 90% Ill.	10% Pel. 90% Ill.	10% Pel. 10% Mont 80% Sand	10% Pel. 10% Mont 80% Sand
5	22.9	28.1	16.5	18.4
10	23.4	22.1	15.4	15.9
15	22.5	20.2	15.3	16.1
20	20.1	18.6	14.5	15.6

TABLE 15

ACTIVATION ENERGIES CALCULATED BY DUPONT SOFTWARE FOR 10% INCREMENTS OF REACTION.

% R'n	100 % Pel	10 % Pel 90% Sand	10% Pel. 80% Sand 10% Kaol	10% Pel. 45% Sand 45% Kaol	10% Pel. 90% Kaol
10	31.9	22.1	25.7	27.1	23.2
20	37.1	24.8	24.4	27.3	23.7
30	42.0	28.2	26.2	28.6	25.1
40	46.5	31.9	29.6	30.0	27.8
50	49.3	35.5	33.0	31.5	28.2

% R'n	10%Pel 80% Sand 10% Mont	10% Pel 80% Sand 10% Mont Repeat	10% Pel 45% Sand 45% Mont	10% Pel 90% Mont
10	30.8	28.4	26.2	21.7
20	31.9	28.8	27.9	22.0
30	34.0	31.2	29.5	23.2
40	37.2	34.1	32.2	24.8
50	39.9	37.0	34.4	26.9

% R'n	10% Pel 80% Sand 10% Ill.	10% Pel 45% Sand 45% Ill.	10% Pel 90% Ill.
10	27.0	25.1	29.4
20	28.9	26.5	28.1
30	32.3	28.5	28.4
40	36.3	31.3	29.6
50	39.8	33.4	31.2

TABLE 16

100% PELICAN RESID - NO INORGANIC SUPPORT: CALCULATION OF ACTIVATION ENERGIES AT EACH 10% REACTION INCREMENT, CORRELATION COEFFICIENTS, AND INTERCEPTS.

% Reaction	Correlation Coeff.	Activation Energy	Intercept
10	-0.998	34.36	23.523
20	-0.999	41.20	28.006
30	-0.998	48.05	32.513
40	-0.999	51.42	34.542
50	-0.999	53.03	35.353
60	-0.999	53.61	35.419
70	-0.999	55.08	36.024
80	-0.999	54.88	35.360
90	-0.999	54.40	34.164

TABLE 17

10% PELICAN RESID ON SAND (90%): CALCULATION OF ACTIVATION ENERGY FOR EACH 10% REACTION INCREMENT , CORRELATION COEFFICIENT, AND INTERCEPT.

% Reaction	Correlation Coeff.	Activ. Energy	Intercept
10	-0.973	41.03	29.426
20	-0.981	42.82	29.721
30	-0.990	51.97	35.590
40	-0.989	56.21	37.950
50	-0.994	56.69	37.71
60	-0.989	62.67	41.43
70	-0.985	68.45	44.856
80	-0.988	70.31	45.85
90	-0.984	74.30	46.978

TABLE 18

REPEAT TGA RUNS WITH 10% PELICAN RESID ON SAND (90%): CALCULATION OF ACTIVATION ENERGIES FOR EACH 10% REACTION INCREMENT, CORRELATION COEFFICIENT, AND INTERCEPT.

% Reaction	Correlation Coeff.	Activation Energy	Intercept
10	-0.997	37.60	27.116
20	-0.995	43.62	30.622
30	-0.998	48.50	33.342
40	-0.952	57.14	39.066
50	-0.990	61.63	41.640
60	-0.989	67.11	44.956
70	-0.989	69.96	46.354
80	-0.990	73.40	48.006
90	-0.986	76.54	48.967

TABLE 19

10% PELICAN RESID ON KAOLIN (10%) AND SAND (80%): DUPLICATE SET OF RUNS: CALCULATION OF ACTIVATION ENERGIES AT EACH 10% REACTION INCREMENT, CORRELATION COEFFICIENTS AND INTERCEPTS.

% Reaction	Correlation Coeff.	Activation Energy	Intercept
10	-0.995	25.41	17.227
20	-0.997	23.70	15.303
30	-0.999	30.60	20.151
40	-0.953	35.69	23.483
50	-0.999	39.68	26.252
60	-0.999	40.46	27.250
70	-0.999	43.14	28.118
80	-0.999	43.20	27.729
90	-0.999	45.50	28.520

TABLE 20

10 % PELICAN RESID ON KAOLIN (45 %) AND SAND (45 %): CALULATION OF ACTIVATION ENERGIES AT EACH 10 % REACTION INCREMENT, CORRELATION COEFFICIENTS AND INTERCEPTS.

% Reaction	Correlation Coeff.	Activation Energy	Intercept
10	-0.995	24.16	16.145
20	-0.988	29.27	19.506
30	-0.997	31.81	20.919
40	-0.999	36.76	24.141
50	-0.997	37.57	24.434
60	-0.996	38.85	25.028
70	-0.998	41.75	26.710
80	-0.998	43.71	27.589
90	-0.999	51.42	31.957

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TABLE 21

10% PELICAN ON KAOLIN (90%): CALCULATION OF ACTIVATION ENERGY FOR EACH 10% REACTION INCREMENT, CORRELATION COEFFICIENT , AND INTERCEPT.

% Reaction	Correlation Coeff.	Activ. Energy	Intercept
10	-0.997	18.62	12.597
20	-0.999	25.06	17.419
30	-0.950	17.35	10.720
40	-0.999	28.35	18.989
50	-0.998	26.67	17.382
60	-0.999	31.45	20.598
70	-0.991	32.45	20.978
80	-0.998	38.13	24.493
90	-0.756	38.75	23.794

TABLE 22

10 % PELICAN RESID ON MONTMORILLONITE (10 %) AND SAND (80 %): CALCULATION OF ACTIVATION ENERGIES AT EACH 10 % REACTION INCREMENT, CORRELATION COEFFICIENTS, AND INTERCEPTS.

% Reaction	Correlation Coeff.	Activation Energy	Intercept
10	-0.988	28.67	20.356
20	-0.997	34.73	24.324
30	-0.995	31.37	21.070
40	-0.999	42.01	28.490
50	-0.999	42.30	28.845
60	-0.999	46.12	30.745
70	-0.998	48.28	31.827
80	-0.999	46.77	30.287
90	-0.999	51.22	32.458

TABLE 23

10% PELICAN RESID ON MONTMORILLONITE (10%) AND SAND (80%): DUPLICATE SET OF RUNS: CALCULATION OF ACTIVATION ENERGIES AT EACH 10 % REACTION INCREMENT, CORRELATION COEFFICIENTS AND INTERCEPTS.

% Reaction	Correlation Coeff.	Activation Energy	Intercept
10	-0.988	25.29	17.555
20	-0.997	31.41	21.656
30	-0.999	34.20	23.125
40	-0.999	38.23	25.664
50	-0.999	41.15	27.425
60	-0.999	42.26	27.900
70	-0.999	44.56	29.138
80	-0.999	44.65	28.725
90	-0.999	46.75	29.320

TABLE 24

10% PELICAN RESID ON 45% MONTMORILLONITE AND 45 % SAND: CALCULATION OF ACTIVATION ENERGIES AT EACH 10% REACTION INCREMENT, CORRELATION COEFFICIENTS AND INTERCEPTS.

% Reaction	Correlation Coeff.	Activation Energy	Intercept
10	-0.995	24.16	16.145
20	-0.998	29.27	19.506
30	-0.997	31.81	20.92
40	-0.999	36.76	24.141
50	-0.997	37.57	24.43
60	-0.996	38.85	25.03
70	-0.998	41.75	26.71
80	-0.998	43.71	27.589
90	-0.999	51.42	31.957

TABLE 25

10% PELICAN RESID. ON MONTMORILLONITE (90 %):CALCULATION OF ACTIVATION ENERGY FOR EACH 10% REACTION INCREMENT, CORRELATION COEFFICIENT, AND INTERCEPT.

% Reaction	Correlation Coeff.	Activation Energy	Intercept
10	-0.988	23.21	16.44
20	-0.990	24.28	16.74
30	-0.957	12.38	6.659
40	-0.999	29.90	20.178
50	-0.999	32.15	21.496
60	-0.999	34.38	22.770
70	-0.999	36.16	23.663
80	-0.999	41.55	26.901
90	-0.728	36.24	22.09

TABLE 26

10 % PELICAN RESID ON ILLITE (10 %) AND SAND (80 %): CALCULATION OF ACTIVATION ENERGIES AT EACH 10 % REACTION INCREMENT, CORRELATION COEFFICIENTS AND INTERCEPTS.

% Reaction	Correlation Coeff.	Activation Energy	Intercept
10	-0.999	26.31	18.465
20	-0.999	32.59	22.663
30	-0.989	31.14	20.783
40	-0.999	40.38	27.271
50	-0.999	42.86	28.767
60	-0.998	43.71	29.039
70	-0.999	47.55	31.354
80	-0.999	49.75	32.365
90	-0.999	51.48	32.643

TABLE 27

10% PELICAN RESID ON ILLITE (45%) AND SAND (45%): CALCULATION OF ACTIVATION ENERGIES AT EACH 10% REACTION INCREMENT, CORRELATION COEFFICIENTS AND INTERCEPTS.

% Reaction	Correlation Coeff.	Activation Energy	Intercept
10	-0.999	22.66	14.857
20	-0.996	28.46	18.946
30	-0.999	25.29	15.939
40	-0.999	32.61	21.086
50	-0.999	36.46	23.603
60	-0.999	35.53	22.629
70	-0.999	37.80	23.915
80	-0.999	42.63	26.838
90	-0.999	45.09	27.623

TABLE 28

10 % PELICAN RESID AND ILLITE (90%): CALCULATION OF ACTIVATION ENERGIES AT EACH 10% REACTION INCREMENT, CORRELATION COEFFICIENTS, AND INTERCEPTS.

% Reaction	Correlation Coeff.	Activation Energy	Intercept
10	-0.975	24.60	17.525
20	-0.999	26.76	18.476
30	-0.999	30.28	20.642
40	-0.999	31.49	21.092
50	-0.999	33.60	22.349
60	-0.998	34.81	22.929
70	-0.999	39.10	25.615
80	-0.999	42.70	27.612
90	-0.998	52.04	33.039

TABLE 29

ACTIVATION ENERGIES FOR MASS LOSS FOR SAMPLES EXAMINED, DETERMINED BY THE METHOD OF FRIEDMAN.

	Activation Energies, Kcal/mole				
% R'n	100% Pel	10% Pel 90% Sand	10% Pel 80% Sand 10% Kaol	10% Pel 45% Sand 45% Kaol	10% Pel 90% Kaol
10	34.4	41.03	25.4	24.2	18.6
20	41.2	42.8	23.7	29.3	25.1
30	48.05	52.0	30.6	31.8	17.4
40	51.4	56.2	35.7	36.8	28.4
50	53.0	56.7	39.7	37.6	26.7
60	53.6	62.7	40.5	38.9	31.5
70	55.1	68.5	43.1	41.8	32.5
80	54.9	70.3	43.2	43.7	38.1
90	54.4	74.3	45.5	51.4	38.8

			10% Pel 80% Sand 10% Mont	10% Pel 45% Sand 45% Mont	10% Pel 90% Mont
10			28.7	24.2	23.2
20			34.7	29.3	24.3
30			31.4	31.8	12.4
40			42.0	36.8	29.9
50			42.3	37.7	32.2
60			46.1	38.9	34.4
70			48.3	41.8	36.2
80			46.8	43.7	41.6
90			51.2	51.4	36.2

TABLE 29 (Cont.)

ACTIVATION ENERGIES FOR MASS LOSS FOR SAMPLES EXAMINED, CALCULATED BY THE METHOD OF FRIEDMAN.

	Activation Energies, Kcal/mole		
% R'n	10% Pel 80% Sand 10% Ill.	10% Pel 45% Sand 45% Ill.	10% Pel 90% Ill.
10	26.3	22.7	24.6
20	32.6	28.5	26.8
30	31.1	25.3	30.3
40	40.4	32.6	31.5
50	42.9	36.5	33.6
60	43.7	35.5	34.8
70	47.6	37.8	39.1
80	49.8	42.6	42.7
90	51.5	45.1	52.0

TABLE 30

SMOOTHED ACTIVATION ENERGIES FOR MASS LOSS FOR SAMPLES EXAMINED, CALCULATED BY METHOD OF FRIEDMAN.

Activation Energies, Kcal/mole

% R'n	100% Pel	10% Pel 90% Sand	10% Pel 80% Sand 10% Kaol	10% Pel 45% Sand 45% Kaol	10% Pel 90% Kaol
10	34.4	38.5	21.5	24.0	16.0
20	41.2	45.0	27.0	29.0	20.5
30	48.1	51.0	32.0	33.0	24.0
40	51.4	56.0	36.0	39.0	27.0
50	53.0	60.0	39.5	41.0	29.5
60	54.0	63.5	42.0	42.5	32.0
70	55.1	67.0	43.5	43.0	34.5
80	55.1	70.5	44.5	44.0	36.5
90	55.1	74.0	45.0	46.0	38.5

	10% Pel 80% Sand 10% Mont	10% Pel 80% Sand 10% Mont Repeat	10% Pel 45% Sand 45% Mont	10% Pel 90% Mont
10	28.7	26.5	24.0	21.5
20	33.0	30.5	29.0	25.0
30	37.0	34.5	33.0	27.5
40	40.5	38.5	36.0	30.0
50	43.5	41.5	38.5	32.0
60	46.0	43.5	40.5	34.5
70	48.0	45.0	42.5	36.0
80	49.5	46.0	44.0	38.5
90	50.5	48.0	45.5	40.0

TABLE 30 (Cont.)

SMOOTHED ACTIVATION ENERGIES FOR MASS LOSS FOR SAMPLES EXAMINED, CALCULATED BY METHOD OF FRIEDMAN.

% R'n	Activation Energies, Kcal/mole 10% Pel 80% Sand 10% Ill	10% Pel 45% Sand 45% Ill	10% Pel 90% Ill	10% Pel 90% Ill
10	26.3	24.0	27.5	23.0
20	32.0	27.0	31.5	27.0
30	36.5	30.0	33.5	30.0
40	40.2	32.5	36.0	32.0
50	43.0	33.5	39.0	34.0
60	45.0	35.5	42.0	36.5
70	48.0	38.5	46.0	40.0
80	49.5	43.0	49.5	42.5
90	51.0	46.5	52.5	47.5

TABLE 31

ACTIVATION ENERGIES BY FRIEDMAN'S METHOD: DUPLICATE RUNS WITH 10% PELICAN RESID. AND 90% SAND.

	Activation Energies	
% Reaction	Run #1	Run #2
10	41.0	37.6
20	42.8	43.6
30	52.0	48.5
40	56.2	57.1
50	56.7	61.6
60	62.7	67.1
70	68.5	70.0
80	70.3	73.4
90	74.3	76.5

TABLE 32

ACTIVATION ENERGIES BY FRIEDMAN'S METHOD: DUPLICATE RUNS WITH 10% PELICAN RESID, 10% MONTMORILLONITE AND 80% SAND

	Activation Energies	
% Reaction	Run #1	Run #2
10	28.7	25.3
20	34.7	31.4
30	31.4	34.2
40	42.0	38.2
50	42.3	41.2
60	46.1	42.3
70	48.3	44.6
80	46.8	44.7
90	51.2	46.8

TABLE 33 TGA INVESTIGATION OF COKE DEVOLATILIZATION

Isothermal Reactor Run #	Sample Type	Temp/ Time C/h	% Coke		% Devolatilization			
					400-525°C		400-600°C	
			Bulk	TGA	5°C/min	15°C/min	5°C/min	15°C/min
52	Pel/Illite/Sand	420/8	1.16	1.10	n.a.	4.40	n.a.	11.09
54	Pel/Illite/Sand	400/8	1.18	1.67	n.a.	3.89	n.a.	8.56
				2.43	n.a.	4.40	n.a.	9.30
60	Pel/Illite/Sand	410/8	0.98	0.92	n.a.	3.79	n.a.	11.60
				0.97	n.a.	1.76	n.a.	6.31
72	Del/Illite/Sand	400/8	2.13	2.99	n.a.		n.a.	9.53
				4.40	n.a.	4.84	n.a.	9.61
80	Del/Illite/Sand	420/4	3.05	3.77	n.a.	2.65	n.a.	6.82
				5.56	n.a.	3.42	n.a.	7.89
79	Pel/Illite/Sand	435/8	2.03	2.12	n.a.	1.46	n.a.	5.52
43	Pel/Mont/Sand	420/8	1.0	0.79	1.13	18.66	8.45	18.97
				0.75				
34	Pel/Sand	420/8	0.68	0.47	0.0	0.0	1.12	0.0
				0.42				
17	Pel/Sand	420/4	0.54	0.22	0.0	0.0	3.0	0.0
				0.31				
54	Pel/Illite/Sand	400/8	1.28	0.76	8.2	2.7	15.0	6.7
				1.14				
47	Pel/Illite/Sand	400/4	0.56	0.82	12.6	7.4	21.2	12.6
				0.88				

TABLE 34

FUEL DEPOSITION ON CLEAN SAND AS A FUNCTION OF TEMPERATURE: BOUSAID AND RAMEY'S RESULTS.

Coking Temperature, C	Fuel Deposition lb/100 lb sand	Atomic H/C Ratio
Original Crude	5.0	1.56
149	4.32	-
260	3.06	-
371	1.57	1.18
427	0.48	0.77
482	0.36	0.70
593	0.33	0.92
649	0.38	0.00

Volume

2

4-9058
2/2

GULF CANADA LIMITED

RESEARCH AND DEVELOPMENT DEPARTMENT
SHERIDAN PARK, ONTARIO, CANADA

INFLUENCE OF THE RESERVOIR MATRIX ON THE MECHANISM AND KINETICS OF THE IN SITU COMBUSTION PROCESS FOR HEAVY OIL RECOVERY

VOLUME II OF II
APPENDICES

by
T. R. Lynch
J. Thomson
G. Cavaliere

ECPease
Reviewed by: ______________________________
E. C. Pease, Project Leader
Heavy Oil

Work on this project was conducted under the auspices of the Canada Centre for Mineral and Energy Technology, Energy, Mines, and Resources Canada

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CANMET SCIENTIFIC AUTHORITY: DR. D. K. FAURSCHOU

GULF CANADA RESEARCH AND DEVELOPMENT DEPARTMENT
JANUARY 1986

MICROMEDIA

48pp

APPENDIX A

TGA DATA FOR THERMAL DECOMPOSITION OF PELICAN 504°C+

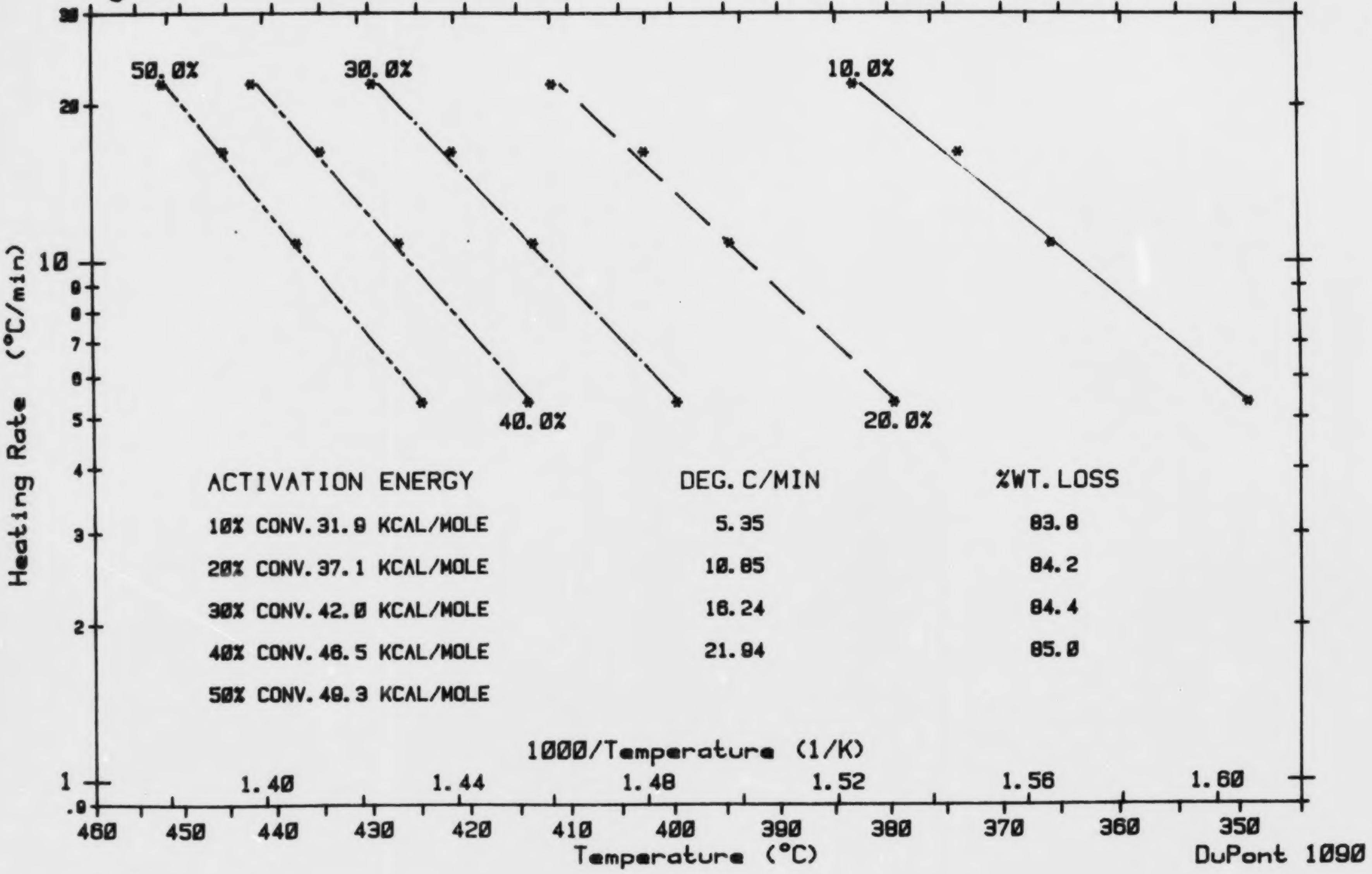
Sample: PEL.RESID.D81-13

Date: 8-Oct-85 Time: 13:54:13

Operator: JT

Program: TGA Kinetics V1.0

Plotted: 8-Oct-85 15:46:06



ACTIVATION ENERGY	DEG. C/MIN	%WT. LOSS
10% CONV. 31.9 KCAL/MOLE	5.35	83.8
20% CONV. 37.1 KCAL/MOLE	10.85	84.2
30% CONV. 42.0 KCAL/MOLE	16.24	84.4
40% CONV. 46.5 KCAL/MOLE	21.84	85.0
50% CONV. 49.3 KCAL/MOLE		

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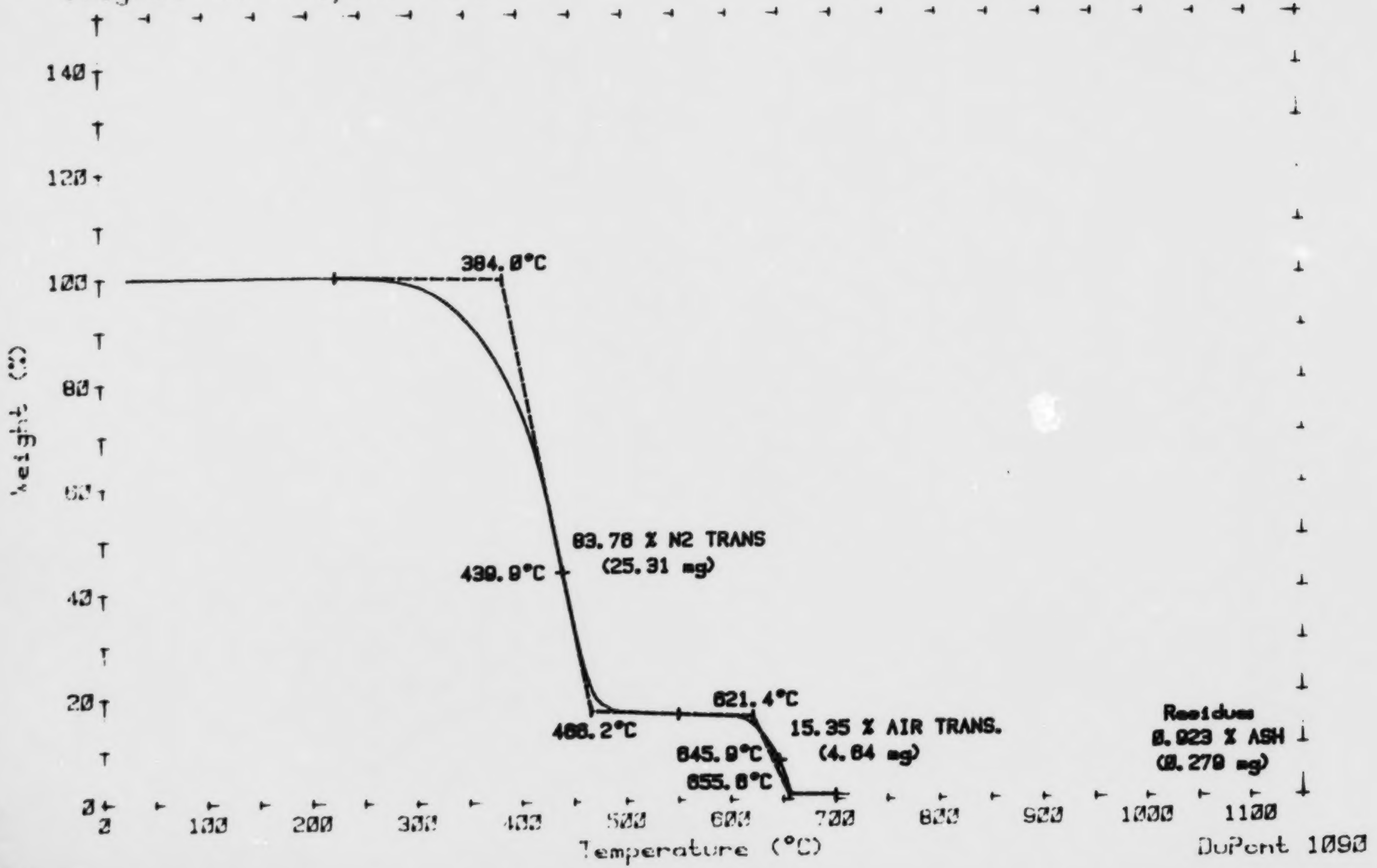
Sample: PEL.RESID.D81-13
Size: 30.22 mg
Rate: 05C/MIN
Program: TGA Analysis V2.0

TGA

Date: 7-Oct-85 Time: 17:08:31
File: PEL/RESID.60 T.LYNCH.08
Operator: JT
Plotted: 8-Oct-85 6:08:11



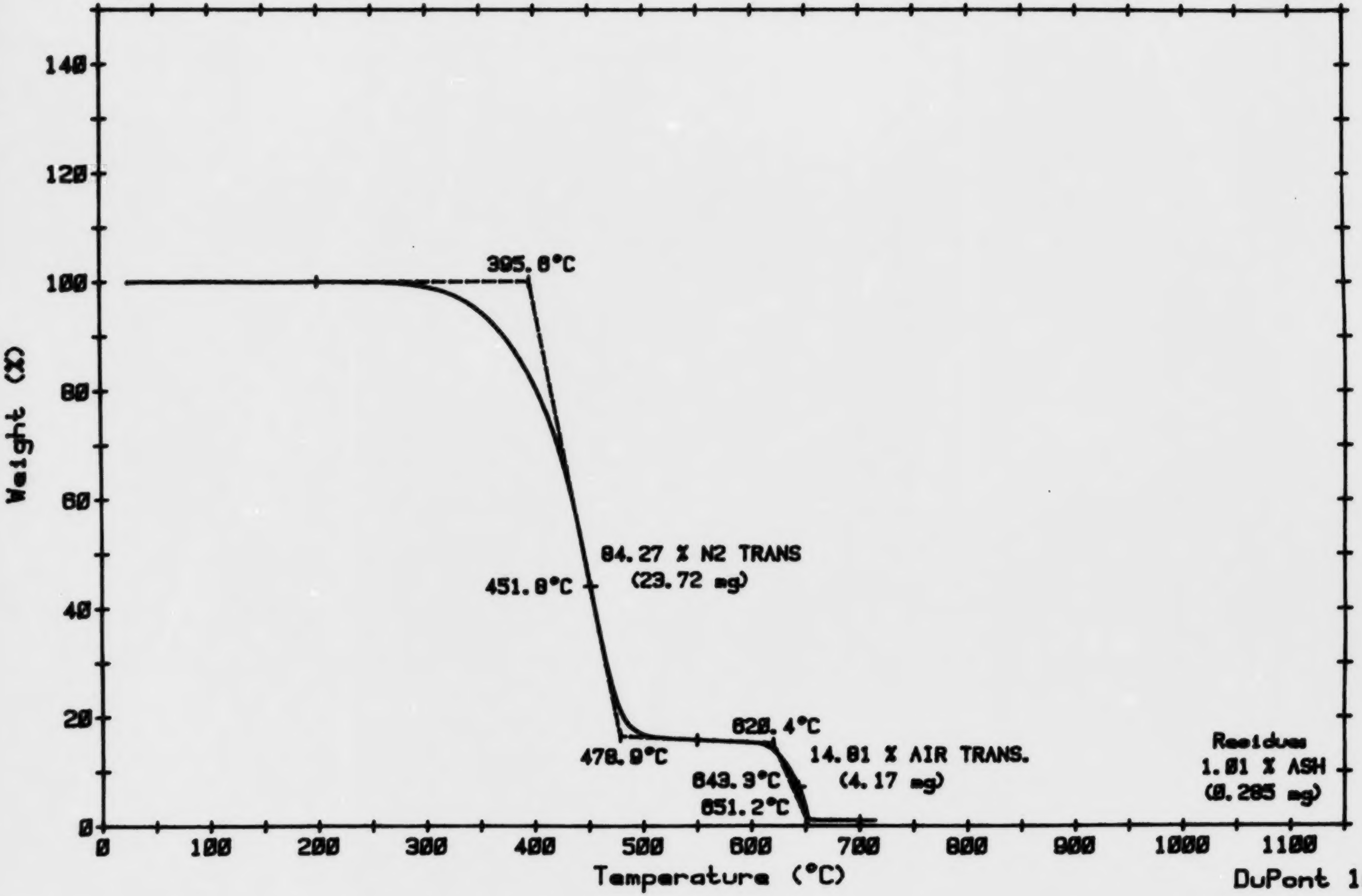
```
Sample: PEL.RESID.D81-13                                    Date:   7-Oct-85    Time: 17:08:31
Size: 30.22 mg                                              File: PEL/RESID.60 T.LYNCH.08
Rate: 05C/MIN                                               Operator: JT
Program: General Analysis V1.0                              Plotted:  9-Oct-85     9:56:07

      Temp       Sig-A      Deriv-A                          Temp       Sig-A      Deriv-A
      [deg]       [%]       [%/min]                          [deg]       [%]       [%/min]

      150.1     100.076       0.00                           250.2      99.887       0.05
      155.3     100.079       0.00                           255.1      99.838       0.06
      160.0     100.076       0.00                           260.1      99.765       0.08
      165.1     100.079       0.00                           265.0      99.686       0.11
      170.1     100.076      -0.02                           270.0      99.583       0.14
      175.2     100.083      -0.03                           275.0      99.461       0.14
      180.2     100.079      -0.01                           280.1      99.322       0.17
      185.2     100.078       0.00                           285.0      99.149       0.20
      190.0     100.083       0.00                           290.2      98.931       0.24
      195.0     100.079      -0.01                           295.1      98.683       0.30
      200.0     100.079       0.01                           300.0      98.405       0.35
      205.1     100.079       0.00                           305.1      98.047       0.41
      210.1     100.068       0.00                           310.2      97.624       0.49
      215.2     100.060       0.01                           315.1      97.157       0.58
      220.2     100.056       0.04                           320.2      96.585       0.65
      225.2     100.036       0.02                           325.1      95.962       0.74
      230.1     100.020       0.01                           330.2      95.231       0.81
      235.2      99.997       0.03                           335.0      94.450       0.91
      240.1      99.970       0.03                           340.1      93.543       0.98
      245.1      99.934       0.05                           345.1      92.550       1.08
                                                             350.2      91.478       1.17
```

```
Sample: PEL.RESID.D81-13                         Date:   7-Oct-85    Time: 17:08:31
Size:  30.22 mg                                  File:  PEL/RESID.60 T.LYNCH.08
Rate:  05C/MIN                                   Operator:  JT
Program:  General Analysis V1.0                  Plotted:   9-Oct-85    10:10:05

     Temp       Sig-A      Deriv-A                   Temp       Sig-A      Deriv-A
     [deg]       [%]       [%/min]                   [deg]       [%]       [%/min]

     350.2     91.478       1.17                     450.0     33.535       4.92
     355.1     90.389       1.26                     455.2     28.912       4.30
     360.0     89.194       1.38                     460.1     25.172       3.55
     365.0     87.828       1.49                     465.2     22.177       2.70
     370.0     86.385       1.59                     470.1     19.890       1.54
     375.1     84.776       1.75                     475.0     18.742       0.86
     380.0     83.138       1.88                     480.1     18.014       0.62
     385.0     81.275       2.04                     485.0     17.527       0.42
     390.1     79.246       2.21                     490.0     17.223       0.24
     395.1     77.088       2.43                     495.1     17.027       0.15
     400.1     74.686       2.62                     500.0     16.892       0.14
     405.2     72.055       2.91                     505.2     16.786       0.09
     410.2     69.119       3.26                     510.1     16.713       0.08
     415.1     65.800       3.71                     515.0     16.650       0.09
     420.1     62.063       4.13                     520.1     16.587       0.06
     425.0     57.903       4.58                     525.1     16.528       0.06
     430.0     53.366       4.97                     530.1     16.475       0.05
     435.0     48.504       5.29                     535.0     16.425       0.04
     440.2     43.182       5.33                     540.1     16.379       0.07
     445.0     38.407       5.22                     545.1     16.328       0.05
                                                     550.0     16.279       0.03
```

Sample: PEL.RESID.D81-13
Size: 28.14 mg
Rate: 10C/MIN
Program: TGA Analysis V2.0

TGA

Date: 8-Oct-85 Time: 8:38:40
File: PEL/RESID.61 T.LYNCH.08
Operator: JT
Plotted: 8-Oct-85 13:57:20



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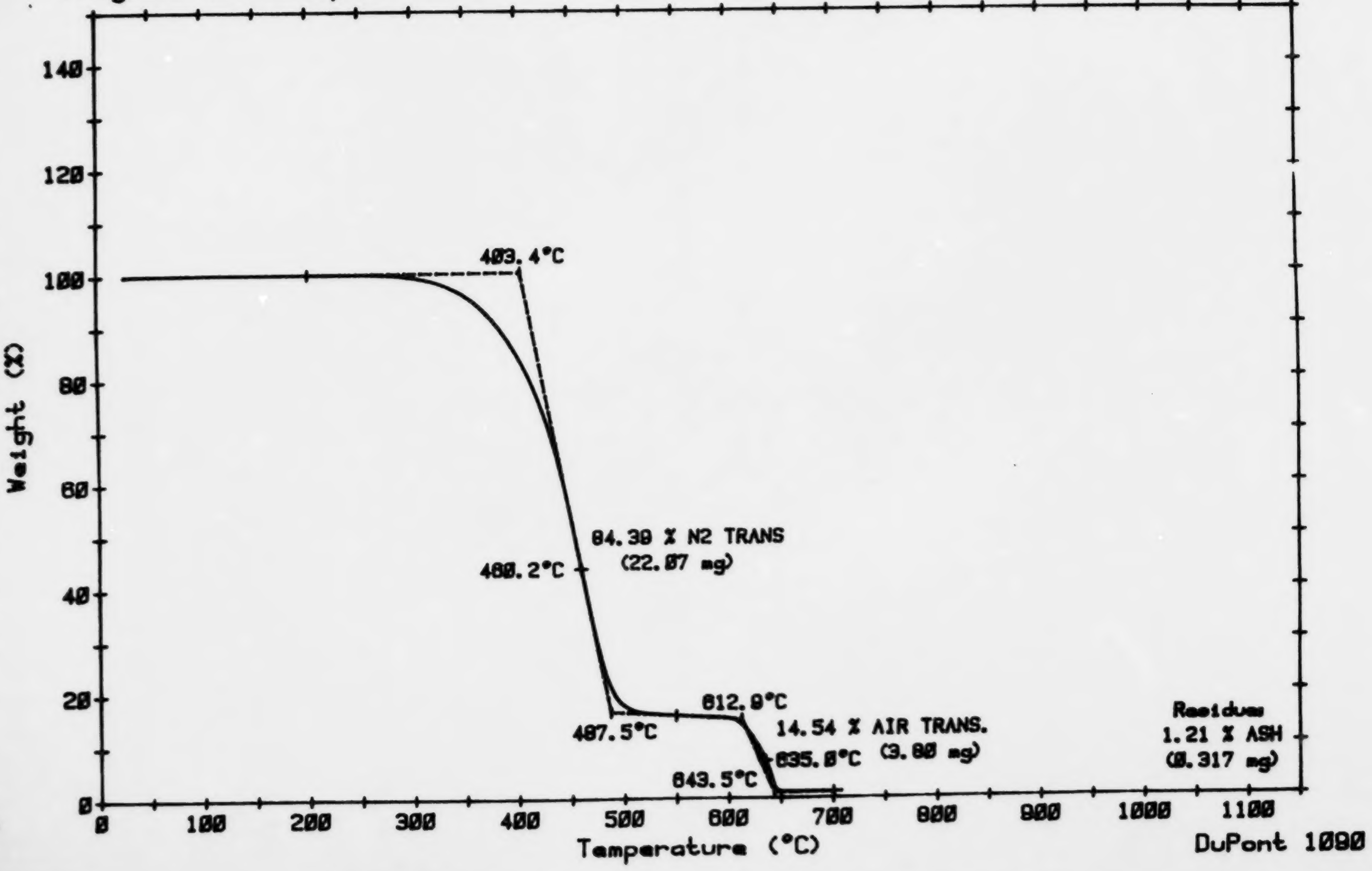
```
Sample: PEL.RESID.D81-13                         Date:   8-Oct-85    Time:   8:39:40
Size: 28.14 mg                                   File:  PEL/RESID.61  T.LYNCH.08
Rate: 10C/MIN                                    Operator:  JT
Program: General Analysis V1.0                   Plotted:   9-Oct-85    10:20:12

      Temp       Sig-A       Deriv-A                  Temp       Sig-A       Deriv-A
      [deg]       [%]        [%/min]                  [deg]       [%]        [%/min]

      150.3     100.046      -0.02                    250.0     100.025       0.04
      155.3     100.053       0.01                    255.0     100.000       0.13
      160.4     100.046       0.02                    260.0      99.979       0.07
      165.4     100.060      -0.03                    265.5      99.929       0.10
      170.4     100.043      -0.02                    270.5      99.869       0.14
      175.4     100.064      -0.07                    275.5      99.805       0.20
      180.4     100.110       0.13                    280.4      99.709       0.22
      185.5     100.117       0.07                    285.3      99.609       0.25
      190.5     100.103       0.00                    290.3      99.481       0.33
      195.0     100.107       0.00                    295.2      99.339       0.34
      200.0     100.110      -0.02                    300.2      99.165       0.40
      205.1     100.110       0.01                    305.1      98.966       0.48
      210.2     100.110       0.00                    310.1      98.732       0.58
      215.3     100.114      -0.01                    315.0      98.444       0.68
      220.3     100.107       0.03                    320.1      98.113       0.80
      225.3     100.096       0.01                    325.0      97.726       0.93
      230.4     100.089       0.02                    330.0      97.275       1.08
      235.4     100.082       0.04                    335.5      96.696       1.24
      240.5     100.080       0.05                    340.4      96.095       1.41
      245.5     100.046       0.06                    345.3      95.409       1.61
                                                      350.1      94.649       1.79
```

```
Sample: PEL.RESID.D81-13                              Date:  8-Oct-85    Time:  8:39:40
Size: 28.14 mg                                        File: PEL/RESID.61 T.LYNCH.08
Rate: 10C/MIN                                         Operator: JT
Program: General Analysis V1.0                        Plotted:  9-Oct-85   10:31:55

      Temp      Sig-A     Deriv-A                        Temp      Sig-A     Deriv-A
      [deg]      [%]      [%/min]                        [deg]      [%]      [%/min]

      350.1     94.849      1.79                         450.2     45.869     10.80
      355.5     93.707      2.01                         455.5     40.257     10.72
      360.5     92.759      2.21                         460.2     35.507     10.30
      365.3     91.725      2.39                         465.0     31.047      9.42
      370.2     90.584      2.67                         470.4     26.617      8.09
      375.2     89.333      2.89                         475.0     23.387      6.19
      380.0     87.978      3.13                         480.1     20.786      4.28
      385.4     86.338      3.41                         485.4     18.935      2.61
      390.3     84.732      3.71                         490.7     17.965      1.83
      395.1     82.985      4.03                         495.1     17.332      1.28
      400.5     80.888      4.42                         500.3     16.842      0.74
      405.3     78.802      4.86                         505.4     16.565      0.43
      410.2     76.507      5.34                         510.0     16.401      0.27
      415.0     73.973      5.95                         515.2     16.284      0.23
      420.3     70.832      6.65                         520.2     16.181      0.17
      425.1     67.670      7.42                         525.3     16.110      0.16
      430.4     63.761      8.25                         530.4     16.039      0.09
      435.2     59.878      9.04                         535.0     16.000      0.10
      440.1     55.610      9.82                         540.0     15.947      0.11
      445.4     50.497     10.53                         545.0     15.890      0.10
                                                         550.1     15.838      0.10
```

Sample: PEL.RESID.D81-13
Size: 26.15 mg
Rate: 15C/MIN
Program: TGA Analysis V2.0

TGA

Date: 8-Oct-85 Time: 11:29:25
File: PEL/RESID.62 T.LYNCH.08
Operator: JT
Plotted: 8-Oct-85 14:13:58



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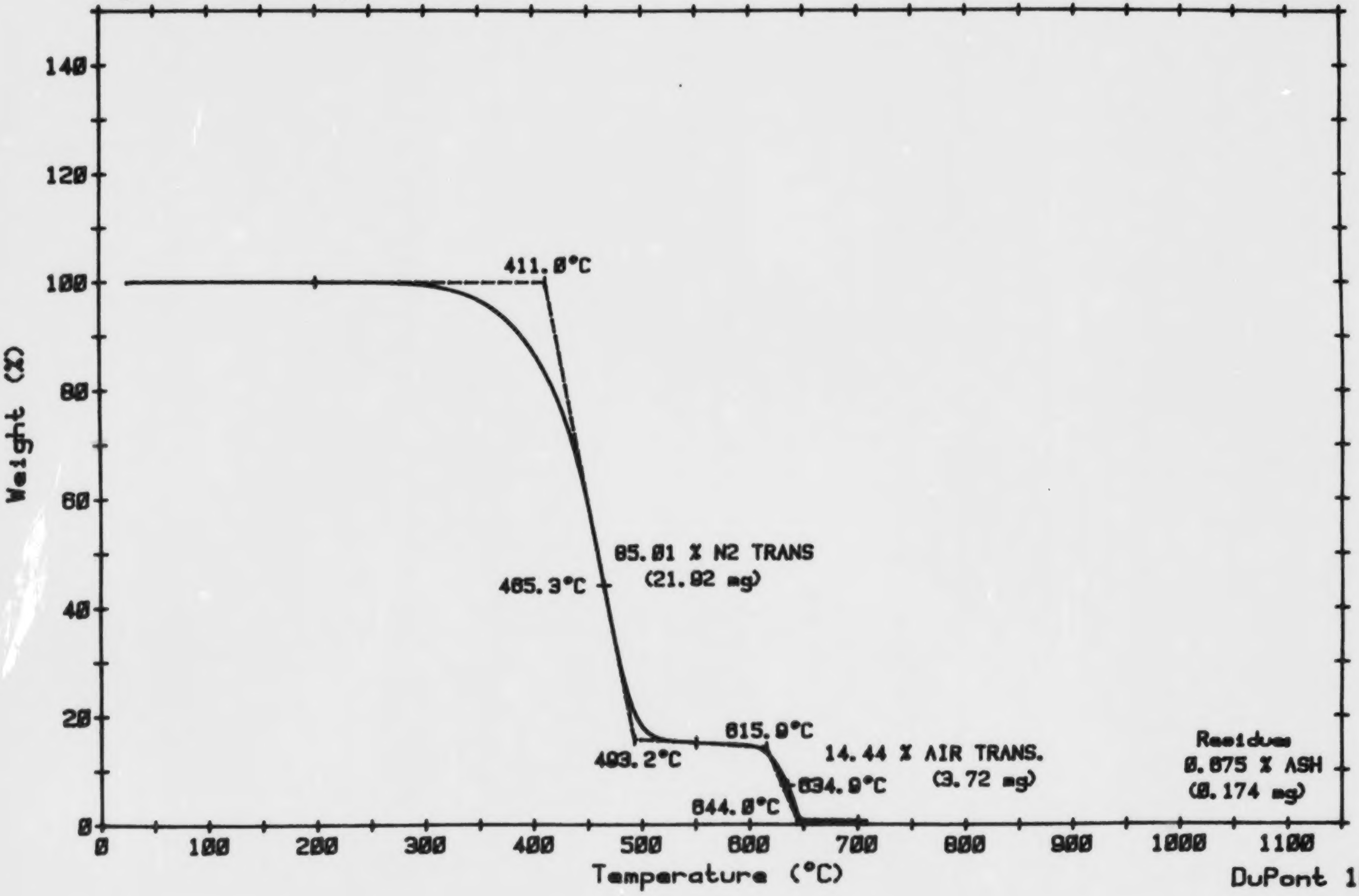
```
Sample: PEL.RESID.D81-13                              Date:  8-Oct-85   Time: 11:29:25
Size: 26.15 mg                                        File: PEL/RESID.62 T.LYNCH.08
Rate: 15C/MIN                                         Operator: JT
Program: General Analysis V1.0                        Plotted:  9-Oct-85   10:51:46

     Temp      Sig-A     Deriv-A                          Temp      Sig-A     Deriv-A
     [deg]      [%]      [%/min]                          [deg]      [%]      [%/min]

     150.2    100.153       0.02                          250.7    100.065       0.06
     155.4    100.145       0.00                          255.8    100.038       0.10
     160.4    100.145       0.01                          260.7    100.011       0.10
     165.5    100.138       0.04                          265.7     99.973       0.14
     170.5    100.141      -0.03                          270.6     99.927       0.18
     175.5    100.134       0.05                          275.6     99.878       0.16
     180.5    100.122      -0.01                          280.5     99.816       0.25
     185.6    100.126       0.01                          285.5     99.736       0.31
     190.7    100.130      -0.03                          290.5     99.637       0.38
     195.7    100.126      -0.01                          295.4     99.518       0.41
     200.7    100.138      -0.02                          300.4     99.381       0.49
     205.8    100.141      -0.01                          305.4     99.224       0.58
     210.0    100.134       0.03                          310.4     99.033       0.70
     215.0    100.134       0.01                          315.3     98.811       0.79
     220.8    100.126       0.01                          320.2     98.547       0.92
     225.0    100.130       0.00                          325.0     98.260       1.05
     230.0    100.122       0.04                          330.0     97.912       1.24
     235.0    100.115       0.02                          335.8     97.434       1.50
     240.0    100.099       0.05                          340.6     96.956       1.69
     245.8    100.080       0.06                          345.6     96.413       1.93
                                                          350.5     95.797       2.18
```

```
Sample: PEL.RESID.D81-13                        Date:   8-Oct-85    Time: 11:29:25
Size: 26.15 mg                                  File: PEL/RESID.62 T.LYNCH.08
Rate: 15C/MIN                                   Operator: JT
Program: General Analysis V1.0                  Plotted:  9-Oct-85    11:17:10

      Temp      Sig-A     Deriv-A                     Temp      Sig-A     Deriv-A
     [deg]       [%]      [%/min]                    [deg]       [%]      [%/min]

     350.5     95.787       2.19                     450.7     53.038      15.10
     355.4     95.105       2.43                     455.5     48.392      15.75
     360.3     94.320       2.72                     460.2     43.608      16.10
     365.1     93.469       3.03                     465.0     38.790      15.79
     370.8     92.344       3.41                     470.6     33.410      14.77
     375.7     91.270       3.74                     475.4     29.181      13.24
     380.5     90.104       4.09                     480.2     25.460      11.36
     385.4     88.819       4.44                     485.6     22.068       8.06
     390.2     87.435       4.82                     490.2     20.030       5.64
     395.0     85.932       5.21                     495.4     18.451       3.57
     400.6     84.020       5.71                     500.7     17.556       2.48
     405.5     82.226       6.27                     505.5     16.963       1.59
     410.4     80.268       6.84                     510.2     16.580       0.89
     415.1     78.104       7.57                     515.6     16.328       0.56
     420.7     75.305       8.47                     520.3     16.191       0.38
     425.5     72.636       9.37                     525.6     16.072       0.33
     430.3     69.680      10.37                     530.3     15.988       0.24
     435.0     66.407      11.43                     535.6     15.908       0.21
     440.5     62.162      12.84                     540.1     15.846       0.18
     445.2     58.139      13.99                     545.5     15.781       0.15
                                                     550.1     15.743       0.17
```

Sample: PEL.RESID.D81-13
Size: 25.79 mg
Rate: 20C/MIN
Program: TGA Analysis V2.0

TGA

Date: 8-Oct-85 Time: 13:54:13
File: PEL/RESID.63 T.LYNCH.08
Operator: JT
Plotted: 8-Oct-85 14:43:57



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```
Sample: PEL.RESID.D81-13                              Date:   8-Oct-85    Time: 13:54:13
Size: 25.79 mg                                        File: PEL/RESID.63 T.LYNCH.08
Rate: 20C/MIN                                         Operator: JT
Program: General Analysis V1.0                        Plotted:  9-Oct-85    13:53:03

      Temp      Sig-A     Deriv-A                        Temp      Sig-A     Deriv-A
      [deg]      [%]      [%/min]                        [deg]      [%]      [%/min]

      150.0    100.124     -0.03                         250.7    100.105     0.06
      156.1    100.140      0.03                         255.3    100.093     0.09
      161.0    100.120      0.02                         261.1    100.070     0.11
      165.8    100.132     -0.03                         265.7    100.039     0.14
      170.5    100.128      0.00                         270.3    100.012     0.18
      175.2    100.132      0.00                         275.9     99.961     0.25
      181.0    100.128      0.01                         280.4     99.903     0.29
      185.5    100.136     -0.01                         286.0     99.829     0.31
      190.1    100.136     -0.01                         290.5     99.767     0.36
      195.8    100.136      0.00                         296.1     99.651     0.44
      200.3    100.136     -0.01                         300.5     99.562     0.51
      206.0    100.143      0.02                         305.0     99.453     0.60
      210.6    100.132      0.01                         310.6     99.271     0.74
      215.2    100.136     -0.01                         315.1     99.124     0.82
      220.9    100.128      0.01                         320.8     98.899     1.02
      225.4    100.124      0.00                         325.3     98.674     1.18
      230.1    100.132      0.01                         330.8     98.364     1.35
      235.8    100.116      0.02                         335.2     98.077     1.53
      240.4    100.120      0.03                         340.7     97.670     1.77
      246.1    100.120      0.03                         345.2     97.290     2.01
                                                         350.7     96.755     2.33
```

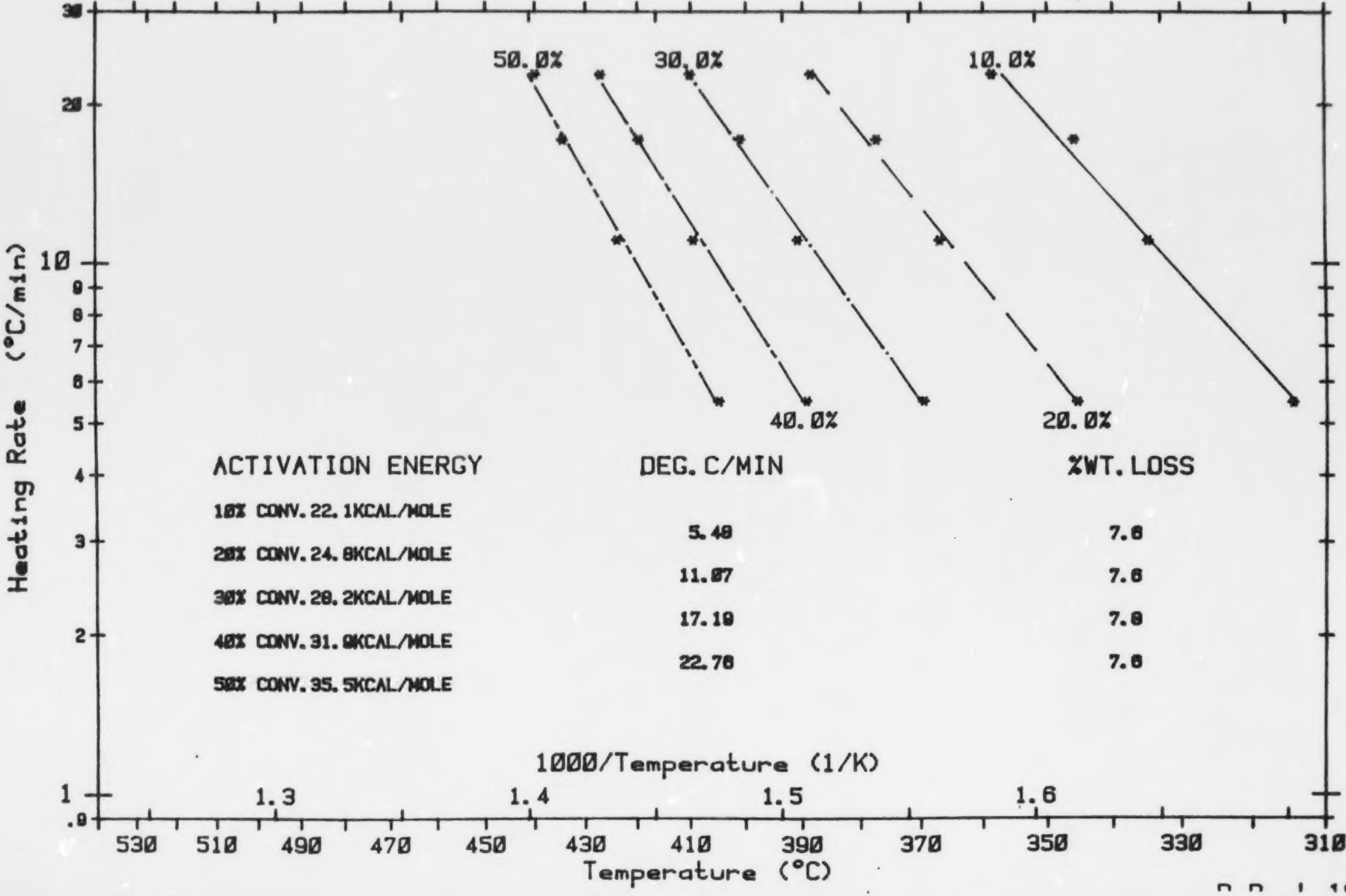
```
Sample: PEL.RESID.D81-13                          Date:   8-Oct-85    Time: 13:54:13
Size:  25.79 mg                                   File:  PEL/RESID.63 T.LYNCH.08
Rate:  20C/MIN                                    Operator:  JT
Program:  General Analysis V1.0                   Plotted:  9-Oct-85    14:30:13

     Temp      Sig-A      Deriv-A                      Temp      Sig-A      Deriv-A
     [deg]      [%]      [%/min]                       [deg]      [%]      [%/min]

     350.7    96.755       2.33                        450.7    58.938      19.25
     355.1    96.282       2.61                        455.9    53.936      20.71
     360.6    95.588       2.96                        460.1    49.666      21.58
     365.1    94.940       3.31                        465.3    44.211      22.01
     370.5    94.058       3.73                        470.6    38.720      21.55
     376.0    93.075       4.16                        476.0    33.463      20.24
     380.4    92.210       4.53                        480.2    29.566      18.45
     385.8    91.004       5.00                        485.6    25.304      15.33
     390.1    89.950       5.48                        490.9    21.943      11.43
     395.5    88.507       6.11                        495.1    19.965       8.60
     400.8    86.898       6.76                        500.1    18.205       5.66
     405.2    85.498       7.35                        505.4    17.073       3.69
     410.6    83.548       8.18                        510.7    16.347       2.29
     415.9    81.411       9.03                        516.0    15.909       1.36
     420.1    79.523       9.88                        520.1    15.682       0.80
     425.5    76.898      11.13                        525.3    15.522       0.60
     430.8    73.959      12.48                        530.4    15.390       0.40
     435.0    71.342      13.82                        535.5    15.320       0.28
     440.3    67.670      15.58                        540.6    15.238       0.27
     445.5    63.536      17.45                        545.6    15.184       0.27
                                                       550.7    15.130       0.20
```

Sample: PEL.RESID.01
90% SAND

Program: TGA Kinetics V1.0

Date: 3-Apr-85 Time: 15:27:55

Operator: TL282-85-82
Plotted: 8-Apr-85 15:41:52

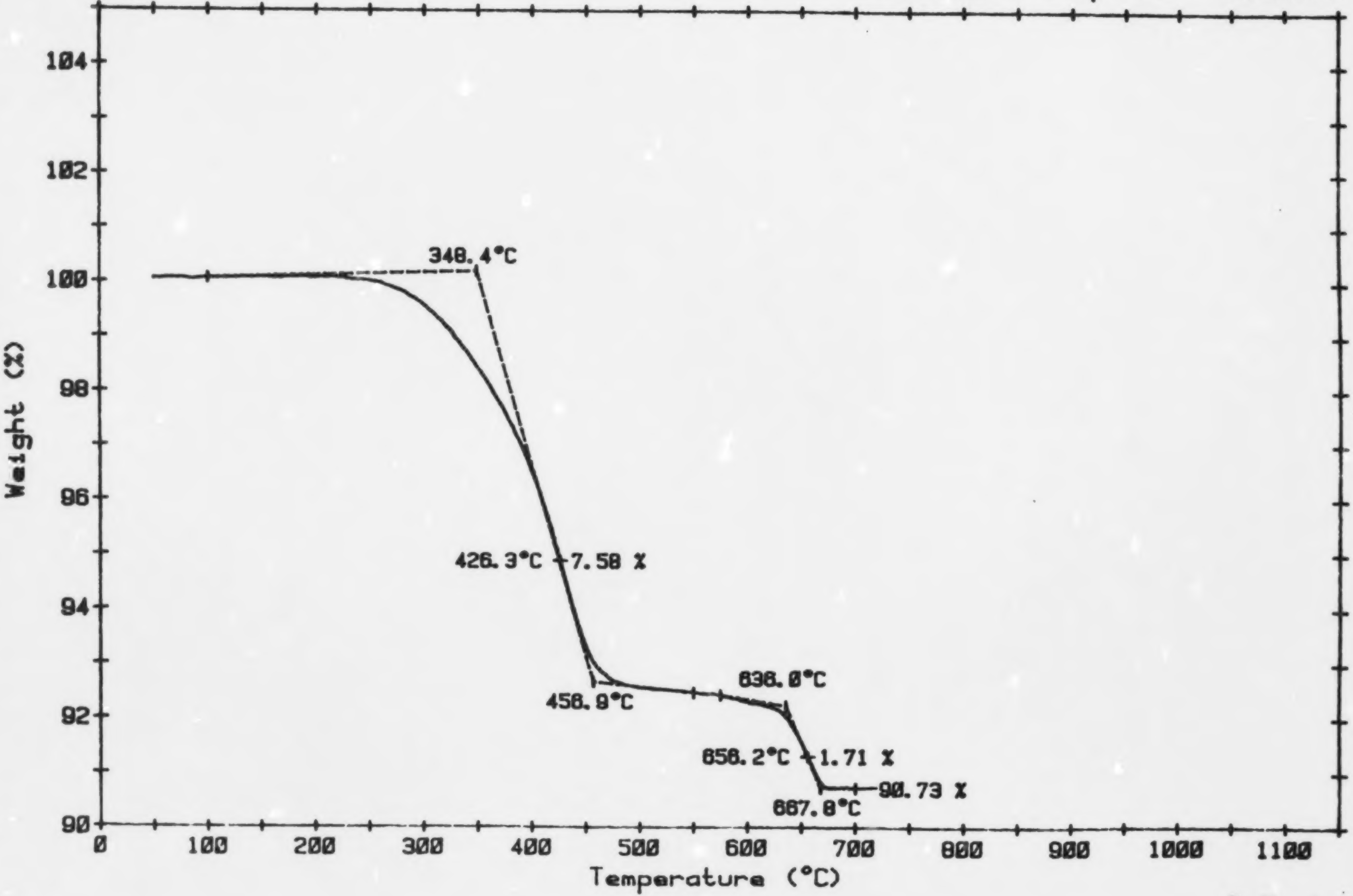


ACTIVATION ENERGY	DEG. C/MIN	%WT. LOSS
10% CONV. 22.1KCAL/MOLE	5.48	7.6
20% CONV. 24.8KCAL/MOLE	11.07	7.6
30% CONV. 28.2KCAL/MOLE	17.19	7.8
40% CONV. 31.9KCAL/MOLE	22.78	7.6
50% CONV. 35.5KCAL/MOLE		

Sample: PEL.RESID.01
Size: 26.72 mg
Rate: 5C/MIN N2 10C/MIN AIR
Program: General Analysis V1.0

TGA

Date: 28-Mar-85 Time: 17:14:37
File: PEL-RESID.02 T.LYNCH.02
Operator: TL282-85-02
Plotted: 4-Apr-85 14:54:23



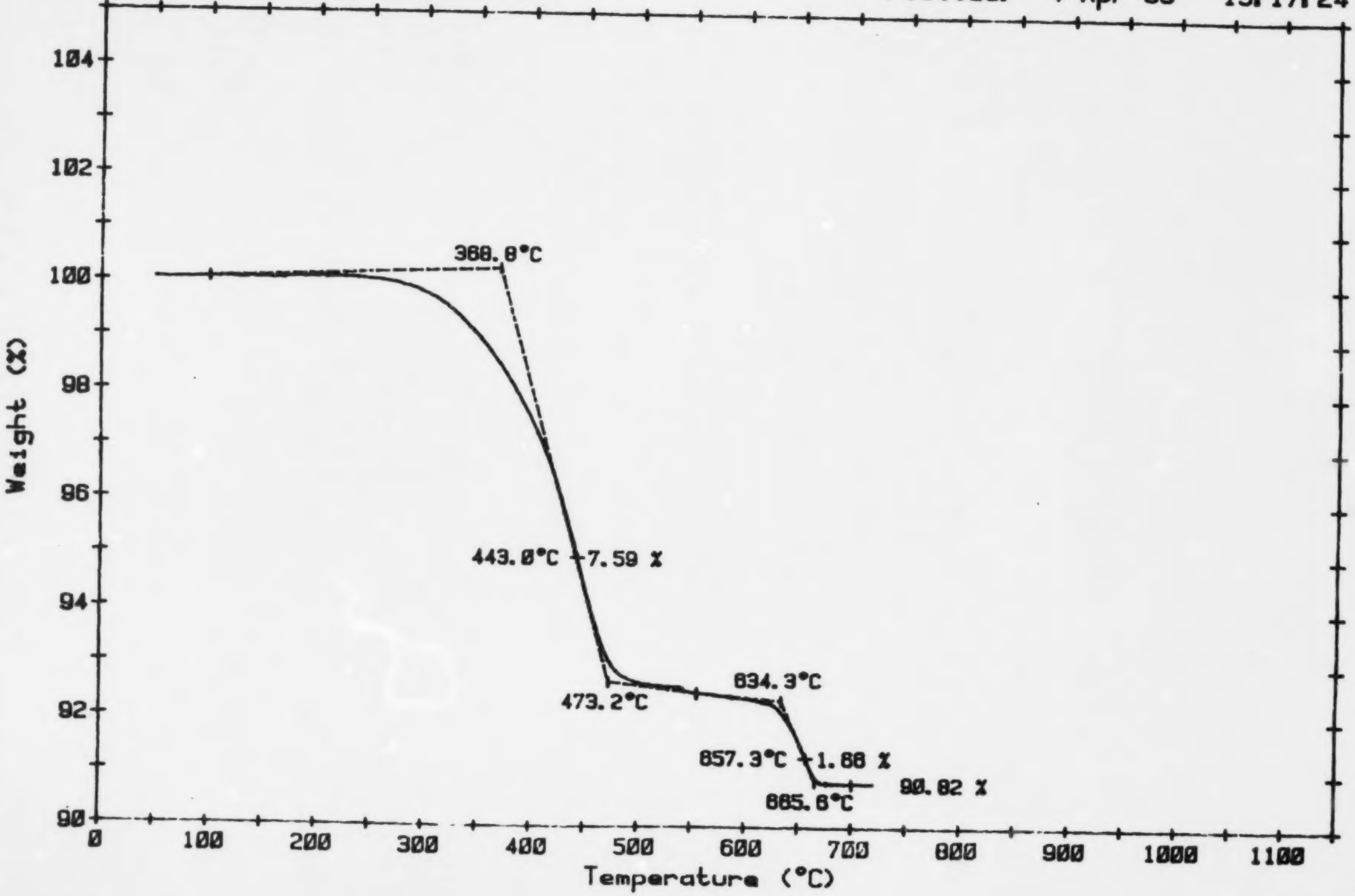
```
Sample: PEL.RESID.01                              Date: 28-Mar-85    Time: 17:14:37
Size: 26.72 mg                                    File: PEL-RESID.02 T.LYNCH.02
Rate: 5C/MIN N2  10C/MIN AIR                      Operator: TL282-85-82
Program: General Analysis V1.0                    Plotted:  4-Apr-85    10:40:29

      Temp       Sig-A       Deriv-A                    Temp       Sig-A       Deriv-A
      [deg]       [%]        [%/min]                    [deg]       [%]        [%/min]

      150.2     100.082        0.00                     250.2     100.034        0.00
      155.0     100.079        0.00                     255.2     100.011        0.01
      160.0     100.086        0.00                     260.0      99.993        0.00
      165.1     100.080        0.00                     265.2      99.951        0.01
      170.1     100.082        0.00                     270.0      99.910        0.01
      175.0     100.082        0.00                     275.1      99.877        0.01
      180.2     100.086        0.00                     280.1      99.835        0.02
      185.2     100.080        0.00                     285.1      99.787        0.02
      190.1     100.101        0.00                     290.1      99.723        0.01
      195.2     100.079        0.00                     295.1      99.671        0.02
      200.0     100.086        0.00                     300.1      99.596        0.02
      205.1     100.080       -0.01                     305.0      99.506        0.02
      210.0     100.082        0.00                     310.0      99.413        0.03
      215.0     100.086       -0.01                     315.1      99.315        0.02
      220.2     100.079        0.01                     320.1      99.207        0.04
      225.1     100.082        0.00                     325.0      99.102        0.04
      230.0     100.056        0.00                     330.0      98.971        0.03
      235.1     100.052        0.00                     335.1      98.840        0.04
      240.2     100.041       -0.01                     340.0      98.716        0.04
      245.2     100.045        0.00                     345.2      98.578        0.04
                                                        350.2      98.428        0.03
```

```
Sample: PEL.RESID.01                          Date: 28-Mar-85     Time: 17:14:37
Size: 26.72 mg                                File: PEL-RESID.02 T.LYNCH.02
Rate: 5C/MIN N2  10C/MIN AIR                  Operator: TL282-85-82
Program: General Analysis V1.0                Plotted: 4-Apr-85    10:50:01

    Temp      Sig-A     Deriv-A                   Temp      Sig-A     Deriv-A
    [deg]      [%]      [%/min]                   [deg]      [%]      [%/min]

    350.2     99.428      0.03                    450.1     93.336      0.07
    355.0     99.286      0.04                    455.0     93.119      0.05
    360.1     99.118      0.04                    460.1     92.954      0.03
    365.0     97.953      0.04                    465.1     92.857      0.01
    370.0     97.785      0.04                    470.0     92.788      0.02
    375.1     97.609      0.05                    475.1     92.711      0.01
    380.2     97.399      0.06                    480.2     92.684      0.01
    385.1     97.197      0.05                    485.1     92.638      0.00
    390.2     97.010      0.06                    490.2     92.617      0.00
    395.1     96.774      0.06                    495.2     92.591      0.01
    400.2     96.509      0.07                    500.2     92.578      0.01
    405.1     96.288      0.09                    505.0     92.561      0.00
    410.2     95.955      0.08                    510.2     92.557      0.01
    415.0     95.663      0.09                    515.2     92.542      0.00
    420.3     95.323      0.10                    520.1     92.557      0.00
    425.2     94.958      0.09                    525.1     92.531      0.00
    430.2     94.597      0.09                    530.1     92.524      0.00
    435.0     94.248      0.09                    535.1     92.520      0.00
    440.0     93.916      0.09                    540.1     92.509      0.00
    445.1     93.598      0.08                    545.2     92.512      0.00
                                                  550.1     92.486      0.00
```

Sample: PEL.RESID.01
Size: 26.59 mg
Rate: 10C/MIN N2 10C/MIN AIR
Program: General Analysis V1.0

TGA

Date: 29-Mar-85 Time: 8:53:17
File: PEL-RESID.03 T.LYNCH.02
Operator: TL282-85-82
Plotted: 4-Apr-85 15:17:24



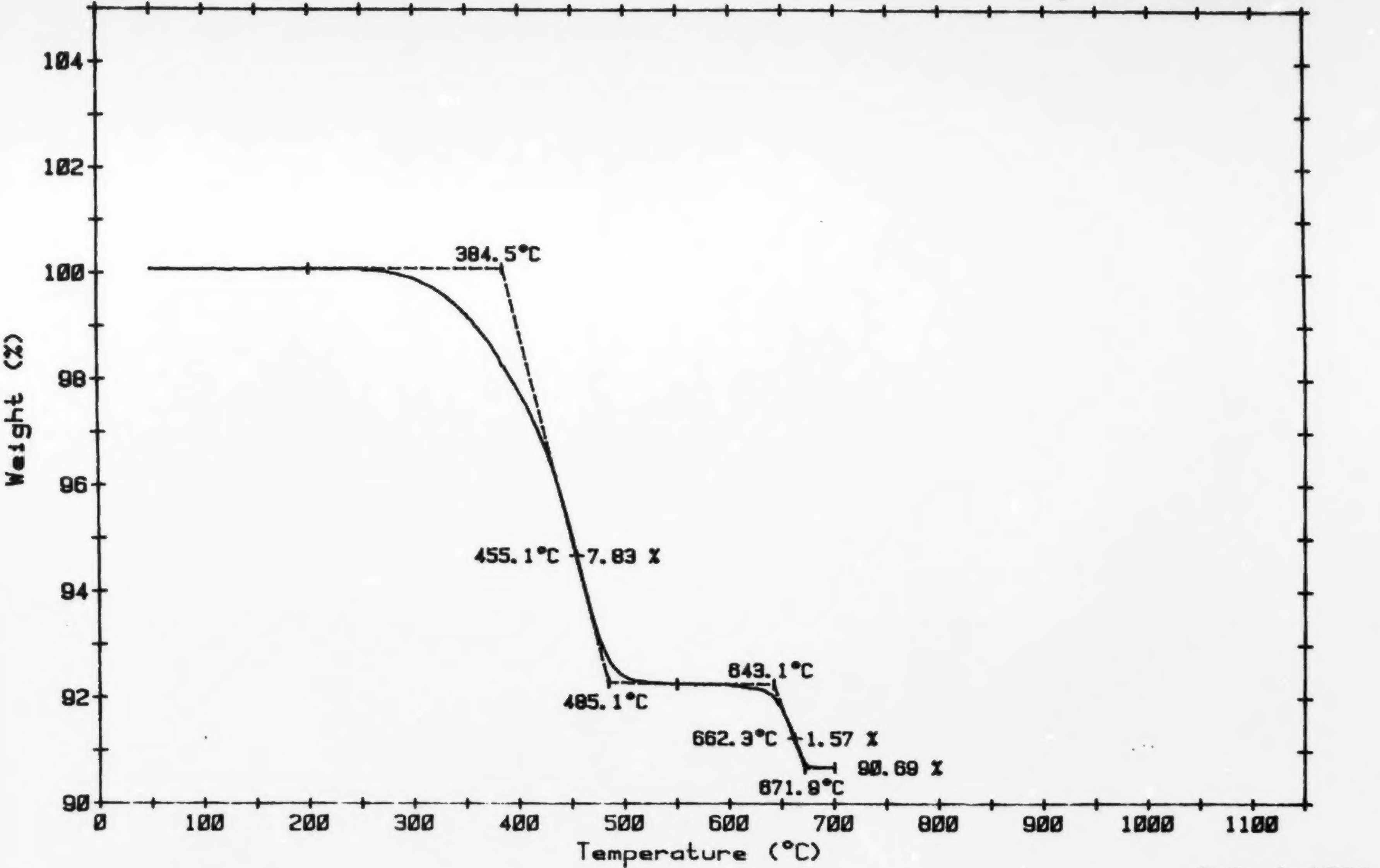
```
Sample: PEL.RESID.01                                    Date: 29-Mar-85     Time:  8:53:17
Size: 26.59 mg                                          File: PEL-RESID.03 T.LYNCH.02
Rate: 10C/MIN N2 10C/MIN AIR                            Operator: TL282-85-82
Program: General Analysis V1.0                          Plotted:  4-Apr-85    11:12:58

       Temp        Sig-A       Deriv-A                        Temp        Sig-A       Deriv-A
       [deg]        [%]        [%/min]                        [deg]        [%]        [%/min]

       150.4     100.053        0.00                          250.5     100.049        0.00
       155.0     100.068       -0.01                          255.0     100.041        0.02
       160.3     100.056        0.00                          260.2     100.023        0.01
       165.0     100.049        0.01                          265.4     100.008        0.02
       170.5     100.079        0.00                          270.0      99.996        0.02
       175.2     100.075       -0.02                          275.2      99.974        0.03
       180.5     100.064       -0.01                          280.4      99.951        0.02
       185.1     100.086        0.01                          285.0      99.921        0.04
       190.4     100.086        0.00                          290.1      99.887        0.04
       195.3     100.071        0.00                          295.3      99.842        0.05
       200.2     100.071       -0.01                          300.5      99.793        0.05
       205.5     100.086        0.01                          305.2      99.759        0.06
       210.3     100.079       -0.01                          310.4      99.703        0.06
       215.0     100.090        0.00                          315.5      99.628        0.08
       220.4     100.083        0.00                          320.3      99.571        0.10
       225.0     100.079        0.01                          325.0      99.496        0.09
       230.2     100.075        0.01                          330.2      99.402        0.10
       235.4     100.071        0.01                          335.3      99.304        0.11
       240.1     100.068        0.02                          340.5      99.191        0.12
       245.4     100.060        0.01                          345.3      99.097        0.13
                                                              350.4      98.988        0.14
```

```
Sample: PEL.RESID.01                              Date: 29-Mar-85    Time:  8:53:17
Size: 26.59 mg                                    File: PEL-RESID.03 T.LYNCH.02
Rate: 10C/MIN N2 10C/MIN AIR                      Operator: TL282-85-82
Program: General Analysis V1.0                    Plotted:  4-Apr-85    11:39:58

      Temp      Sig-A     Deriv-A                       Temp      Sig-A     Deriv-A
      [deg]      [%]      [%/min]                       [deg]      [%]      [%/min]

      350.4     98.988      0.14                        450.4     94.381      0.39
      355.5     98.864      0.14                        455.2     94.035      0.37
      360.1     98.740      0.15                        460.0     93.727      0.34
      365.3     98.608      0.17                        465.2     93.418      0.28
      370.4     98.466      0.15                        470.4     93.159      0.22
      375.0     98.330      0.16                        475.2     92.997      0.16
      380.1     98.168      0.18                        480.0     92.873      0.11
      385.5     97.980      0.19                        485.2     92.790      0.08
      390.4     97.811      0.19                        490.3     92.734      0.05
      395.4     97.631      0.20                        495.4     92.700      0.04
      400.4     97.435      0.23                        500.5     92.662      0.02
      405.4     97.224      0.25                        505.0     92.640      0.02
      410.0     97.014      0.28                        510.5     92.629      0.01
      415.5     96.747      0.28                        515.3     92.617      0.01
      420.4     96.472      0.32                        520.1     92.617      0.01
      425.3     96.179      0.33                        525.1     92.602      0.02
      430.1     95.870      0.36                        530.4     92.591      0.01
      435.0     95.536      0.39                        535.0     92.583      0.01
      440.4     95.141      0.41                        540.5     92.583      0.01
      445.1     94.772      0.40                        545.5     92.508      0.02
                                                        550.4     92.489      0.02
```

Sample: PEL.RESID.01
Size: 27.91 mg
Rate: 15C/MIN N2 10C/MIN AIR
Program: General Analysis V1.0

TGA

Date: 4-Apr-85 Time: 8:52:20
File: PEL-RESID.07 T.LYNCH.02
Operator: TL282-85-82
Plotted: 4-Apr-85 12:02:40



Du Pont 1090

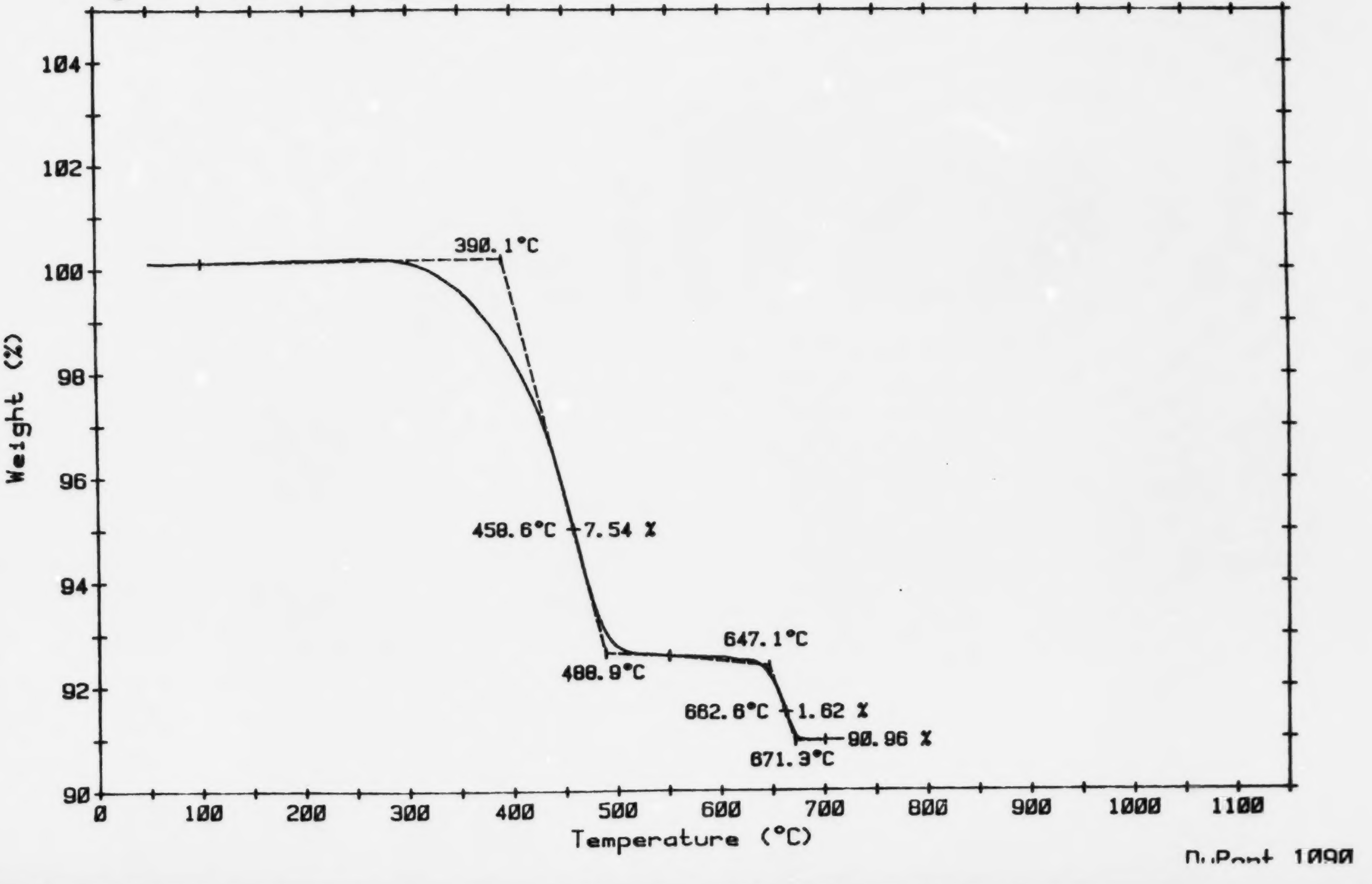
```
Sample: PEL.RESID.01                                      Date:  4-Apr-85   Time:  8:52:
Size: 27.91 mg                                            File: PEL-RESID.07 T.LYNCH.02
Rate: 15C/MIN N2 10C/MIN AIR                              Operator: TL282-85-82
Program: General Analysis V1.0                            Plotted:  4-Apr-85   12:33:20

        Temp       Sig-A      Deriv-A                          Temp       Sig-A      Deriv-A
        [deg]       [%]       [%/min]                          [deg]       [%]       [%/min]

        150.3     100.075      -0.01                           250.7     100.093       0.06
        155.7     100.090       0.00                           255.2     100.084       0.01
        160.0     100.072       0.02                           260.6     100.050       0.00
        165.4     100.079      -0.01                           265.1     100.054       0.01
        170.7     100.079      -0.01                           270.5     100.043       0.02
        175.2     100.082      -0.01                           275.8     100.029       0.04
        180.5     100.082       0.00                           280.2     100.007       0.03
        185.0     100.090       0.00                           285.6      99.989       0.05
        190.5     100.082       0.00                           290.0      99.971       0.04
        195.1     100.080       0.00                           295.2      99.943       0.04
        200.5     100.093       0.01                           300.5      99.910       0.07
        205.9     100.086      -0.01                           305.8      99.864       0.08
        210.5     100.080      -0.01                           310.2      99.824       0.08
        215.0     100.082       0.02                           315.6      99.778       0.09
        220.3     100.082       0.00                           320.1      99.720       0.11
        225.7     100.082       0.00                           325.5      99.649       0.12
        230.2     100.082      -0.01                           330.7      99.570       0.14
        235.6     100.086      -0.01                           335.2      99.495       0.15
        240.1     100.097       0.00                           340.5      99.409       0.16
        245.4     100.100      -0.02                           345.8      99.308       0.17
                                                               350.2      99.226       0.17
```

```
Sample: PEL.RESID.01                                Date:   4-Apr-85    Time:   8:52:20
Size: 27.91 mg                                      File: PEL-RESID.07 T.LYNCH.02
Rate: 15C/MIN N2 10C/MIN AIR                        Operator: TL282-85-82
Program: General Analysis V1.0                      Plotted:   4-Apr-85    12:41:15

      Temp       Sig-A       Deriv-A                     Temp       Sig-A       Deriv-A
      [deg]       [%]        [%/min]                     [deg]       [%]        [%/min]

      350.2      99.226        0.17                      450.0      95.083        0.67
      355.4      99.122        0.20                      455.1      94.676        0.67
      360.5      98.989        0.21                      460.1      94.284        0.66
      365.7      98.860        0.21                      465.1      93.894        0.64
      370.8      98.735        0.22                      470.1      93.532        0.57
      375.2      98.606        0.28                      475.1      93.209        0.49
      380.2      98.441        0.32                      480.1      92.941        0.38
      385.3      98.262        0.23                      485.0      92.740        0.31
      390.4      98.104        0.29                      490.7      92.564        0.18
      395.5      97.932        0.27                      495.5      92.468        0.16
      400.6      97.757        0.31                      500.4      92.410        0.09
      405.8      97.567        0.34                      505.1      92.353        0.05
      410.8      97.355        0.37                      510.7      92.331        0.02
      415.1      97.173        0.38                      515.4      92.321        0.03
      420.4      96.929        0.42                      520.0      92.299        0.01
      425.7      96.685        0.46                      525.4      92.292        0.02
      430.2      96.431        0.51                      530.7      92.285        0.00
      435.4      96.101        0.54                      535.3      92.270        0.02
      440.7      95.761        0.59                      540.6      92.281        0.00
      445.8      95.410        0.64                      545.1      92.253        0.02
                                                         550.5      92.260        0.01
```

Sample: PEL.RESID.01
Size: 26.05 mg
Rate: 20C/MIN N2 10C/MIN AIR
Program: General Analysis V1.0

TGA

Date: 3-Apr-85 Time: 15:27:55
File: PEL-RESID.06 T.LYNCH.02
Operator: TL282-85-82
Plotted: 4-Apr-85 13:42:52



```
Sample: PEL.RESID.01                                    Date:   3-Apr-85    Time: 15:27:55
Size: 26.05 mg                                          File: PEL-RESID.06 T.LYNCH.02
Rate: 20C/MIN N2 10C/MIN AIR                            Operator: TL282-85-82
Program: General Analysis V1.0                          Plotted:  4-Apr-85    13:55:36

      Temp      Sig-A      Deriv-A                    Temp      Sig-A      Deriv-A
      [deg]      [%]       [%/min]                    [deg]      [%]       [%/min]

      150.8    100.142      -0.01                     250.5    100.211      -0.01
      155.7    100.148      -0.01                     255.2    100.203       0.02
      160.7    100.154      -0.01                     261.0    100.188       0.02
      165.8    100.157      -0.02                     265.6    100.188       0.01
      170.7    100.157       0.01                     270.4    100.184       0.01
      175.6    100.157       0.00                     275.3    100.180       0.00
      180.6    100.154      -0.01                     280.2    100.180       0.01
      185.6    100.169      -0.02                     286.1    100.173       0.04
      190.7    100.168      -0.01                     290.8    100.146       0.06
      195.6    100.173       0.00                     295.6    100.127       0.03
      200.6    100.173      -0.01                     300.4    100.119       0.06
      205.5    100.177       0.01                     305.0    100.081       0.07
      210.3    100.161       0.01                     310.6    100.058       0.09
      215.2    100.169      -0.01                     315.4    100.012       0.10
      220.1    100.165      -0.01                     320.0     99.973       0.11
      225.0    100.177      -0.02                     325.7     99.912       0.13
      231.2    100.180      -0.02                     330.3     99.858       0.15
      236.0    100.192      -0.01                     336.1     99.777       0.16
      240.9    100.192      -0.01                     340.6     99.716       0.17
      245.7    100.200      -0.02                     345.2     99.647       0.17
                                                      351.1     99.555       0.21
```

```
Sample: PEL.RESID.01                          Date:   3-Apr-85    Time: 15:27:55
Size: 26.05 mg                                File: PEL-RESID.06 T.LYNCH.02
Rate: 20C/MIN N2 10C/MIN AIR                  Operator: TL282-85-82
Program: General Analysis V1.0                Plotted:  4-Apr-85    14:30:39

     Temp       Sig-A      Deriv-A                 Temp       Sig-A      Deriv-A
    [deg]        [X]       [X/min]                [deg]        [X]       [X/min]

     351.1     99.555        0.21                  451.0     95.596        0.83
     355.8     99.470        0.22                  455.3     95.262        0.85
     360.4     99.370        0.26                  460.8     94.836        0.87
     365.1     99.267        0.27                  465.3     94.487        0.86
     370.7     99.125        0.28                  470.7     94.057        0.80
     375.3     99.009        0.28                  475.2     93.761        0.72
     381.0     98.860        0.31                  480.6     93.419        0.61
     385.6     98.741        0.33                  486.0     93.154        0.45
     390.2     98.595        0.37                  490.4     92.987        0.35
     395.9     98.410        0.38                  495.7     92.851        0.24
     400.3     98.253        0.40                  500.0     92.774        0.16
     406.1     98.057        0.42                  505.3     92.705        0.10
     410.6     97.877        0.46                  510.7     92.671        0.06
     415.1     97.693        0.47                  516.0     92.640        0.05
     420.7     97.451        0.53                  520.3     92.632        0.03
     425.3     97.224        0.58                  525.6     92.617        0.02
     431.0     96.925        0.63                  530.9     92.600        0.02
     435.4     96.660        0.68                  535.1     92.609        0.02
     441.0     96.307        0.74                  540.2     92.590        0.01
     445.4     96.007        0.79                  545.5     92.586        0.01
                                                   550.6     92.582        0.02
```

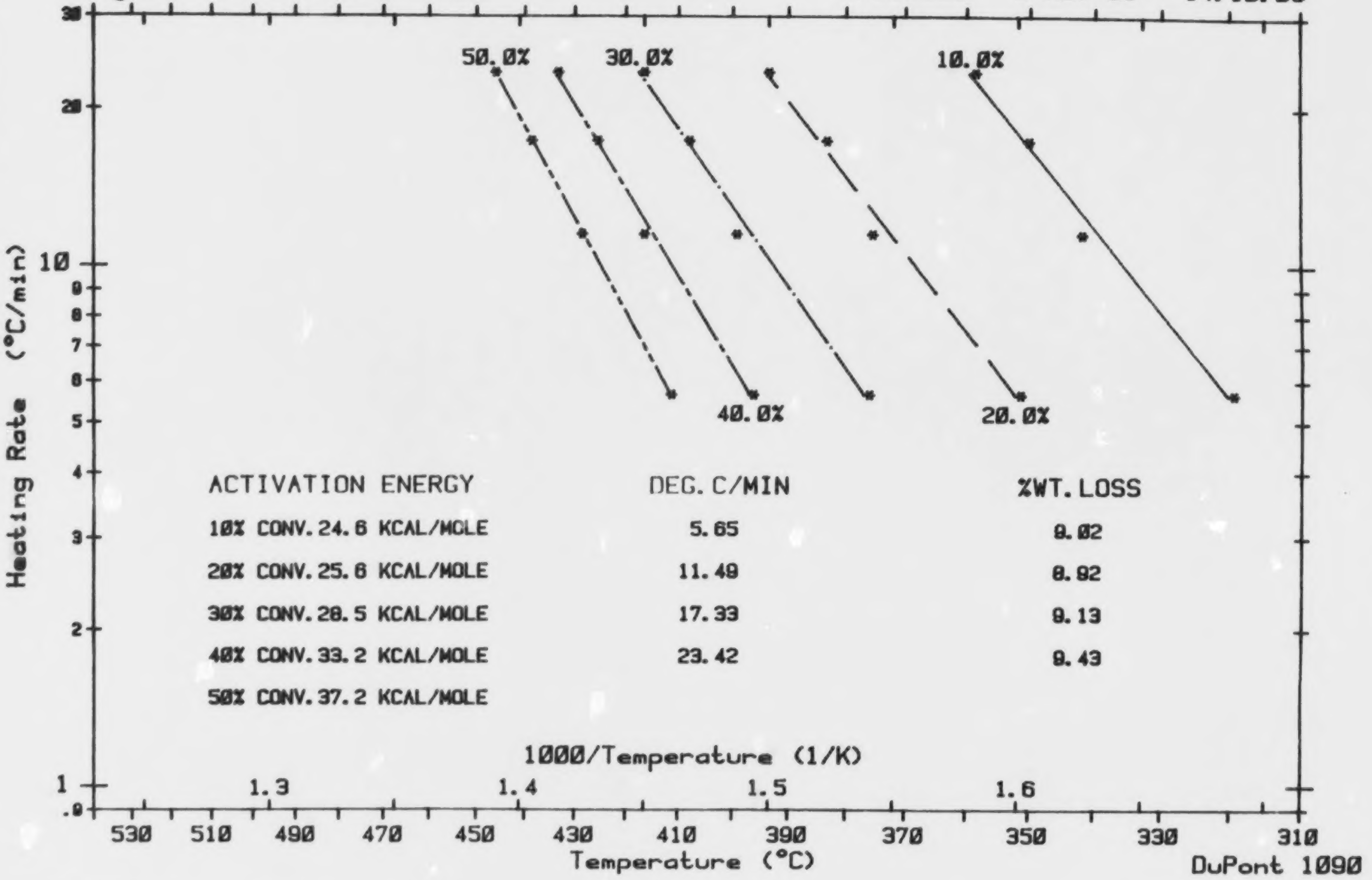
Sample: 90% SAND

Date: 30-Oct-85 Time: 13:18:23

Program: TGA Kinetics V1.0

Operator: JT

Plotted: 1-Nov-85 14:10:05



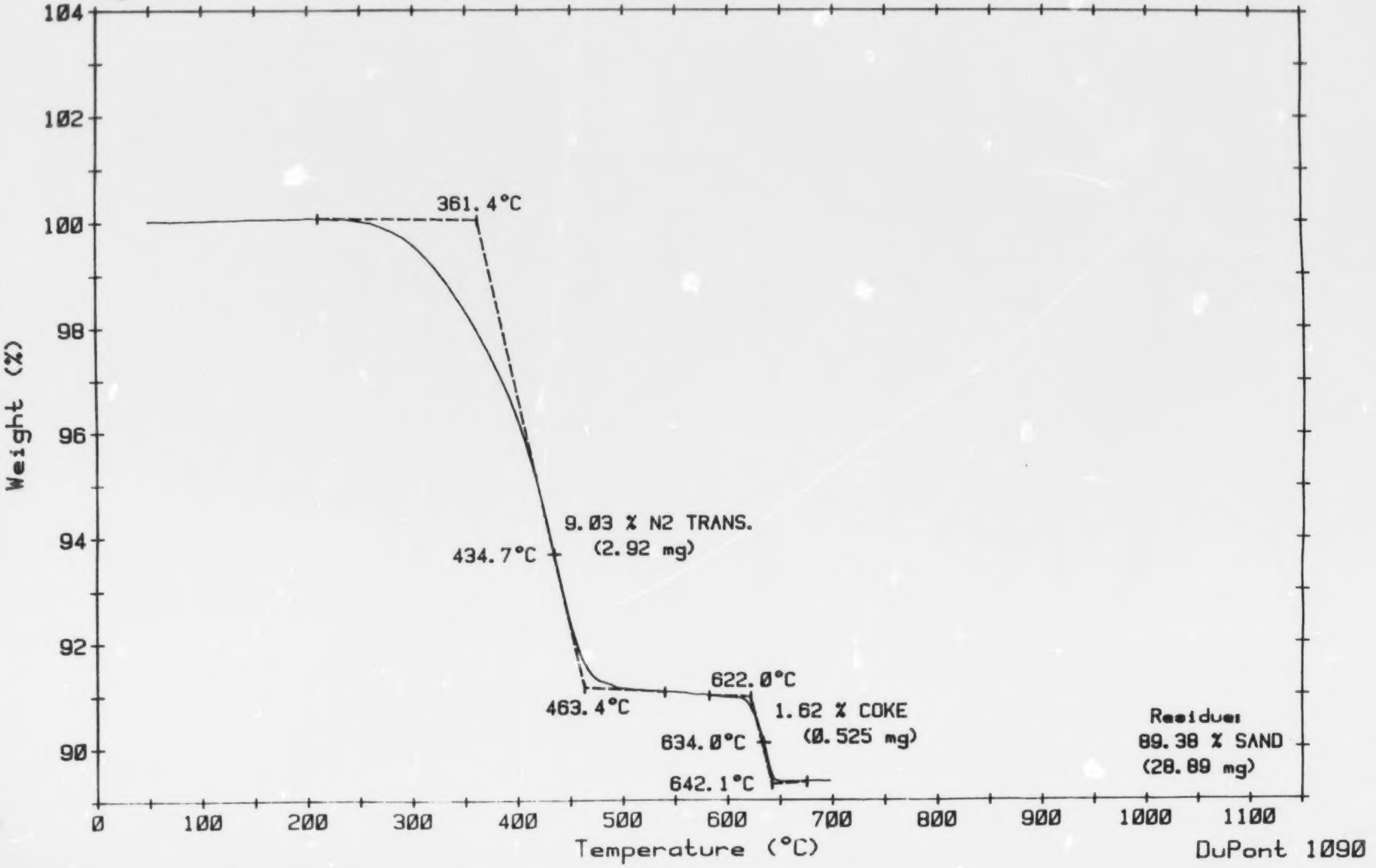
ACTIVATION ENERGY	DEG. C/MIN	%WT. LOSS
10% CONV. 24.6 KCAL/MOLE	5.65	8.02
20% CONV. 25.6 KCAL/MOLE	11.49	8.92
30% CONV. 28.5 KCAL/MOLE	17.33	9.13
40% CONV. 33.2 KCAL/MOLE	23.42	9.43
50% CONV. 37.2 KCAL/MOLE		

DuPont 1090

Sample: 90% SAND
Size: 32.32 mg
Rate: 05C/MIN
Program: TGA Analysis V2.0

TGA

Date: 30-Oct-85 Time: 8:00:10
File: PEL/RES.01 SCRAP BOOK JT
Operator: JT
Plotted: 1-Nov-85 10:26:28



DuPont 1090

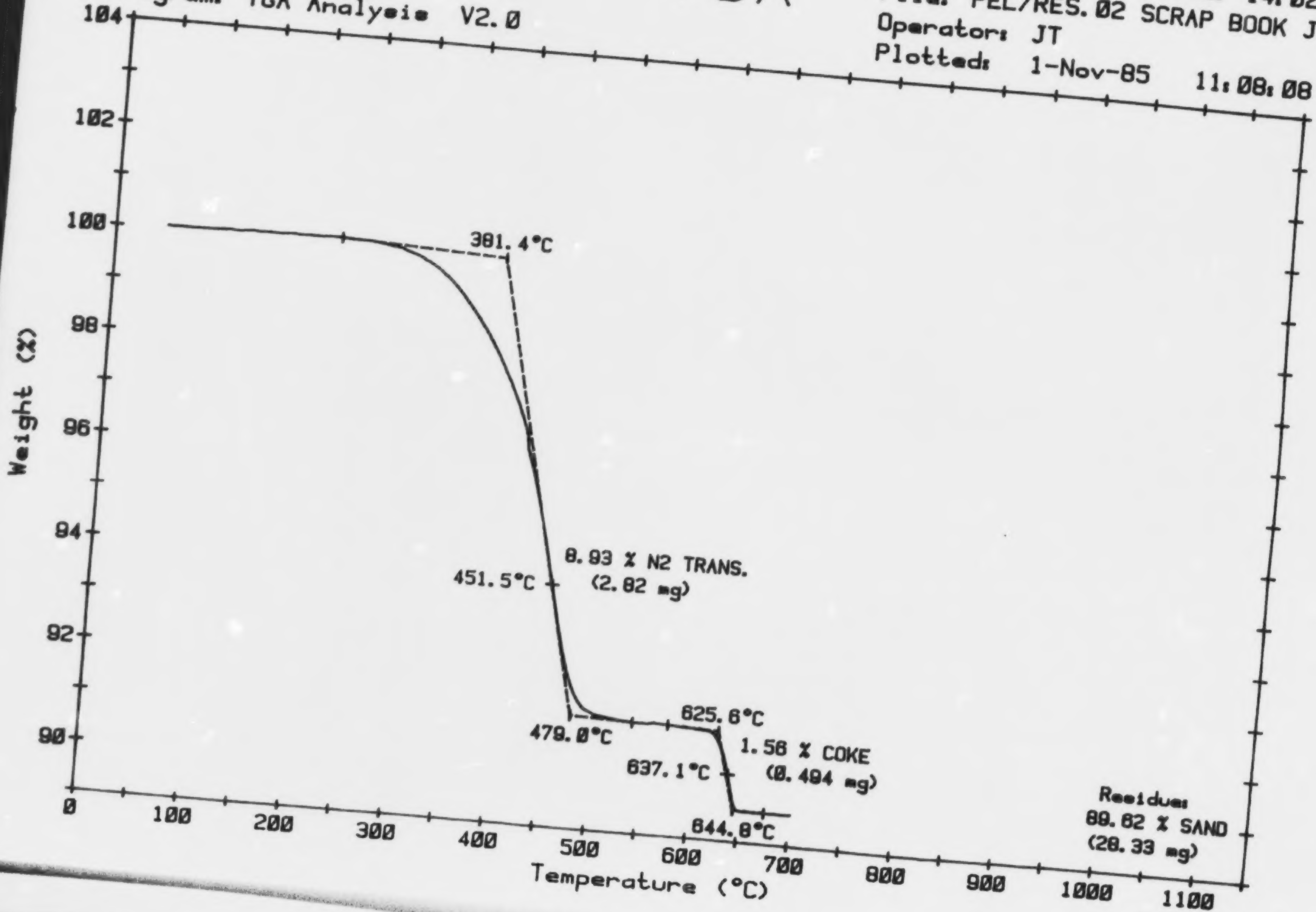
```
Sample: 90% SAND                                   Date: 30-Oct-85    Time:  8:00:10
Size: 32.32 mg                                     File: PEL/RES.01 SCRAP BOOK JT
Rate: 05C/MIN                                      Operator: JT
Program: General Analysis V1.0                     Plotted:  1-Nov-85    12:19:16

     Temp       Sig-A      Deriv-A                     Temp       Sig-A      Deriv-A
     [deg]       [%]       [%/min]                     [deg]       [%]       [%/min]

     150.1     100.056      -0.01                      250.0     100.015      0.01
     155.2     100.053      -0.01                      255.2      99.997      0.00
     160.0     100.056       0.00                      260.1      99.978      0.01
     165.0     100.053       0.00                      265.1      99.935      0.03
     170.1     100.065      -0.01                      270.2      99.898      0.03
     175.2     100.053       0.00                      275.1      99.855      0.03
     180.1     100.049       0.00                      280.1      99.811      0.02
     185.2     100.053      -0.01                      285.2      99.762      0.03
     190.2     100.056      -0.01                      290.2      99.706      0.05
     195.1     100.056      -0.01                      295.3      99.626      0.05
     200.2     100.059      -0.01                      300.1      99.542      0.05
     205.2     100.062       0.00                      305.0      99.462      0.06
     210.1     100.071       0.00                      310.2      99.356      0.06
     215.2     100.065      -0.01                      315.0      99.261      0.07
     220.2     100.059      -0.01                      320.2      99.140      0.07
     225.2     100.059      -0.01                      325.2      99.016      0.07
     230.0     100.043       0.01                      330.1      98.886      0.08
     235.1     100.059       0.02                      335.2      98.741      0.08
     240.0     100.040       0.01                      340.0      98.592      0.09
     245.1     100.028       0.01                      345.1      98.434      0.09
                                                       350.1      98.286      0.09
```

```
Sample: 90% SAND                              Date: 30-Oct-85   Time:  8:00:10
Size: 32.32 mg                                File: PEL/RES.01 SCRAP BOOK JT
Rate: 05C/MIN                                 Operator: JT
Program: General Analysis V1.0                Plotted:  1-Nov-85   12:49:36

     Temp       Sig-A      Deriv-A                 Temp      Sig-A      Deriv-A
     [deg]       [%]       [%/min]                 [deg]      [%]       [%/min]

     350.1      98.286      0.09                   450.1     92.355      0.20
     355.2      98.119      0.09                   455.0     92.033      0.18
     360.0      97.952      0.10                   460.2     91.736      0.14
     365.2      97.766      0.11                   465.2     91.526      0.11
     370.0      97.584      0.10                   470.2     91.374      0.07
     375.1      97.383      0.12                   475.0     91.294      0.04
     380.1      97.175      0.12                   480.1     91.247      0.02
     385.1      96.974      0.12                   485.1     91.210      0.02
     390.0      96.751      0.13                   490.0     91.167      0.03
     395.1      96.504      0.15                   495.2     91.130      0.02
     400.2      96.232      0.16                   500.1     91.120      0.01
     405.2      95.938      0.17                   505.2     91.102      0.01
     410.2      95.613      0.20                   510.0     91.086      0.01
     415.1      95.272      0.21                   515.2     91.083      0.00
     420.0      94.917      0.22                   520.0     91.077      0.00
     425.0      94.508      0.24                   525.2     91.071      0.01
     430.2      94.050      0.26                   530.1     91.062      0.00
     435.0      93.636      0.25                   535.0     91.052      0.01
     440.1      93.184      0.25                   540.1     91.046      0.00
     445.1      92.754      0.24                   545.1     91.043      0.01
                                                   550.2     91.040      0.01
```

Sample: 90% SAND
Size: 31.62 mg
Rate: 10C/MIN
Program: TGA Analysis V2.0

TGA

Date: 31-Oct-85 Time: 14:02:53
File: PEL/RES.02 SCRAP BOOK JT
Operator: JT
Plotted: 1-Nov-85 11:08:08



DuPont 1090

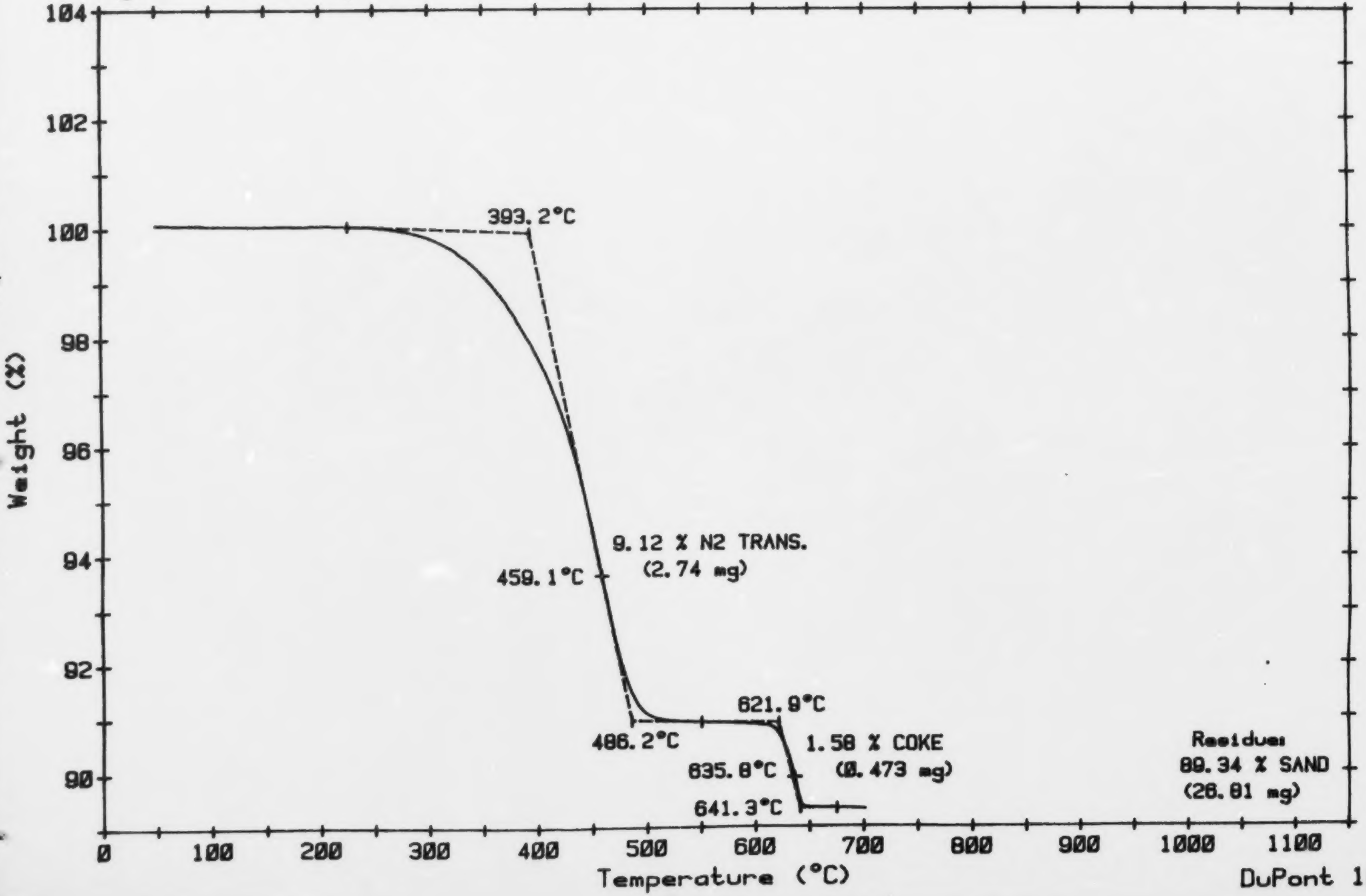
```
Sample: 90% SAND
Size: 31.62 mg                                                Date: 31-Oct-85    Time: 14:02:53
Rate: 10C/MIN                                                 File: PEL/RES.02 SCRAP BOOK JT
Program: General Analysis V1.0                                Operator: JT
                                                              Plotted: 1-Nov-85   13:12:32

   Temp       Sig-A      Deriv-A                      Temp       Sig-A      Deriv-A
   [deg]       [%]       [%/min]                      [deg]       [%]       [%/min]

   150.1     100.089       0.01                       250.2     100.070       0.02
   155.2     100.082      -0.01                       255.4     100.057       0.04
   160.4     100.085      -0.03                       260.0     100.051      -0.01
   165.1     100.079       0.00                       265.3     100.035       0.04
   170.2     100.085      -0.01                       270.4     100.016       0.04
   175.4     100.089       0.03                       275.1     100.003       0.05
   180.0     100.089       0.00                       280.3      99.987       0.10
   185.2     100.089       0.01                       285.4      99.940       0.06
   190.4     100.092      -0.01                       290.1      99.915       0.08
   195.0     100.092       0.01                       295.2      99.877       0.09
   200.2     100.098       0.00                       300.4      99.836       0.13
   205.5     100.095      -0.03                       305.1      99.785       0.10
   210.2     100.098       0.04                       310.4      99.728       0.12
   215.4     100.098       0.00                       315.0      99.677       0.15
   220.0     100.095       0.01                       320.2      99.598       0.20
   225.3     100.092       0.01                       325.4      99.519       0.15
   230.4     100.079      -0.01                       330.1      99.440       0.20
   235.1     100.082       0.02                       335.3      99.339       0.23
   240.3     100.079      -0.02                       340.4      99.228       0.24
   245.0     100.082       0.03                       345.0      99.124       0.27
                                                      350.2      99.004       0.28
```

```
Sample: 90% SAND                                   Date: 31-Oct-85    Time: 14:02:53
Size: 31.62 mg                                     File: PEL/RES.02 SCRAP BOOK JT
Rate: 10C/MIN                                      Operator: JT
Program: General Analysis V1.0                     Plotted:  1-Nov-85    13:27:54

     Temp       Sig-A      Deriv-A                     Temp       Sig-A      Deriv-A
    [deg]        [%]       [%/min]                    [deg]        [%]       [%/min]

     350.2     99.004       0.28                      450.3     93.813       1.03
     355.4     98.865       0.30                      455.4     93.355       1.01
     360.0     98.754       0.31                      460.5     92.896       0.96
     365.3     98.605       0.31                      465.1     92.545       0.86
     370.5     98.460       0.36                      470.1     92.181       0.77
     375.2     98.311       0.37                      475.1     91.884       0.58
     380.4     98.134       0.40                      480.2     91.650       0.43
     385.0     97.969       0.43                      485.2     91.479       0.26
     390.2     97.770       0.45                      490.3     91.369       0.21
     395.4     97.574       0.47                      495.4     91.321       0.09
     400.5     97.350       0.50                      500.5     91.274       0.08
     405.1     97.157       0.55                      505.1     91.252       0.04
     410.3     96.904       0.58                      510.1     91.236       0.05
     415.5     96.628       1.13                      515.1     91.220       0.02
     420.1     96.163       0.70                      520.1     91.207       0.04
     425.3     95.841       0.73                      525.0     91.198       0.05
     430.3     95.480       0.85                      530.5     91.179       0.01
     435.4     95.094       0.88                      535.5     91.176       0.02
     440.5     94.671       0.96                      540.3     91.166      -0.01
     445.2     94.275       1.01                      545.4     91.168      -0.01
                                                      550.4     91.172       0.01
```

Sample: 90% SAND
Size: 30.01 mg
Rate: 15C/MIN
Program: TGA Analysis V2.0

TGA

Date: 29-Oct-85 Time: 8:16:35
File: PEL/RES.03 SCRAP BOOK JT
Operator: JT
Plotted: 1-Nov-85 11:17:49



DuPont 1090

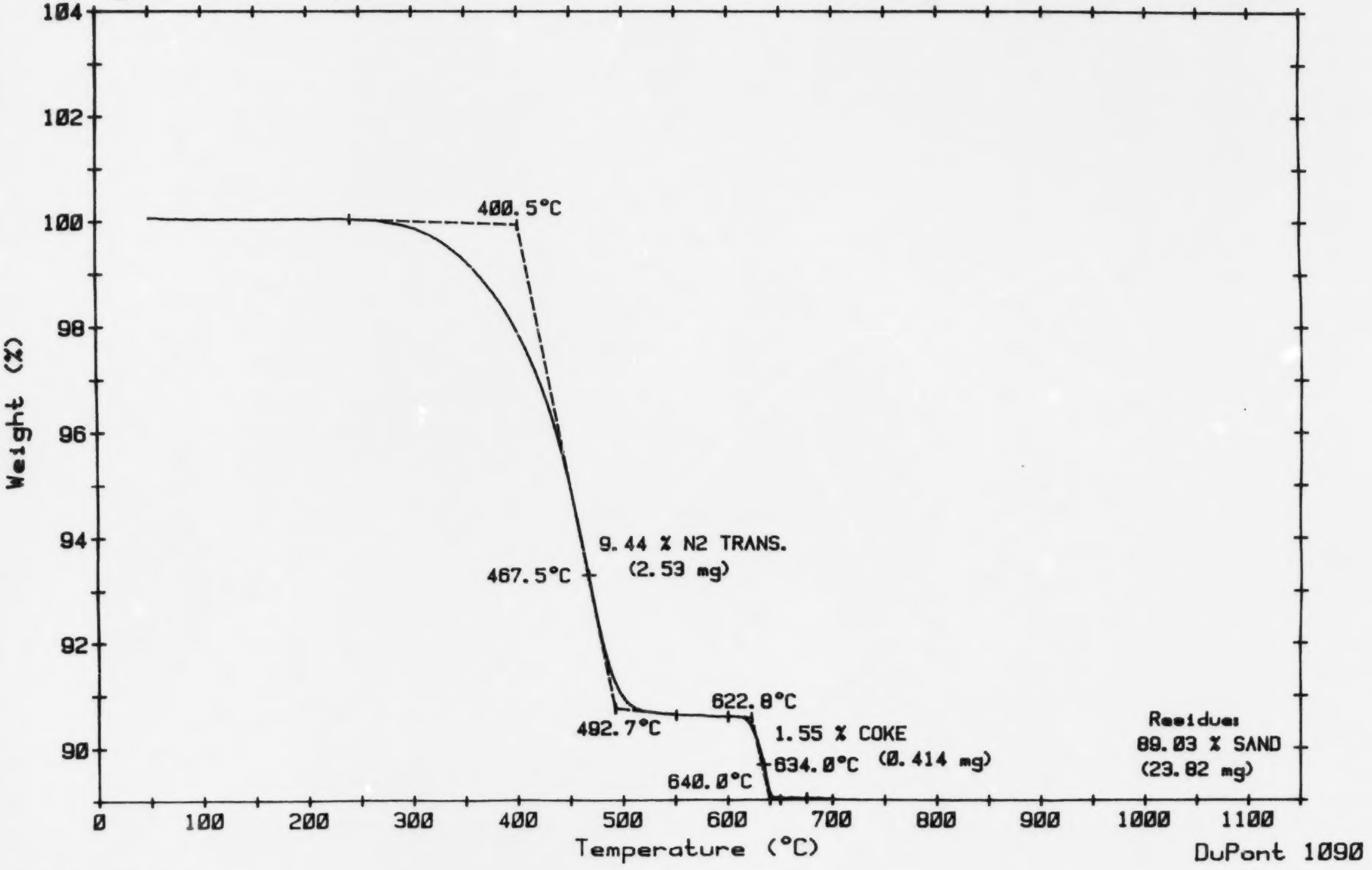
```
Sample: 90% SAND                                    Date: 29-Oct-85   Time:  8:16:35
Size: 30.01 mg                                      File: PEL/RES.03 SCRAP BOOK JT
Rate: 15C/MIN                                       Operator: JT
Program: General Analysis V1.0                      Plotted:  1-Nov-85   13:39:33

      Temp      Sig-A      Deriv-A                      Temp      Sig-A      Deriv-A
     [deg]       [%]       [%/min]                     [deg]       [%]       [%/min]

     150.3     100.043      -0.03                      250.4     100.017       0.04
     155.6     100.040       0.02                      255.7     100.003       0.03
     160.0     100.040       0.00                      260.1      99.990       0.07
     165.2     100.043      -0.01                      265.4      99.970       0.03
     170.4     100.043       0.01                      270.7      99.967       0.06
     175.7     100.037       0.00                      275.1      99.950       0.07
     180.0     100.047      -0.01                      280.3      99.927       0.06
     185.3     100.040       0.00                      285.6      99.903       0.09
     190.6     100.043      -0.03                      290.8      99.880       0.09
     195.8     100.050      -0.02                      295.2      99.857       0.11
     200.2     100.050       0.01                      300.4      99.817       0.15
     205.5     100.053      -0.01                      305.8      99.773       0.13
     210.8     100.043       0.04                      310.1      99.733       0.18
     215.2     100.037      -0.01                      315.4      99.683       0.19
     220.6     100.040       0.01                      320.7      99.620       0.19
     225.8     100.037       0.03                      325.0      99.567       0.27
     230.2     100.030       0.01                      330.2      99.490       0.26
     235.4     100.027       0.02                      335.5      99.404       0.27
     240.7     100.020      -0.01                      340.7      99.317       0.31
     245.1     100.030       0.02                      345.1      99.237       0.34
                                                       350.3      99.127       0.36
```

```
Sample: 90% SAND                                      Date: 29-Oct-85   Time:  8:16:35
Size: 30.01 mg                                        File: PEL/RES.03 SCRAP BOOK JT
Rate: 15C/MIN                                         Operator: JT
Program: General Analysis V1.0                        Plotted:  1-Nov-85   13:47:38

      Temp      Sig-A     Deriv-A                       Temp      Sig-A     Deriv-A
      [deg]      [%]      [%/min]                       [deg]      [%]      [%/min]

      350.3     99.127      0.36                        450.7     94.429      1.62
      355.6     99.014      0.42                        455.8     93.943      1.64
      360.8     98.887      0.43                        460.8     93.440      1.64
      365.1     98.777      0.45                        465.0     93.040      1.62
      370.3     98.644      0.52                        470.1     92.557      1.47
      375.6     98.474      0.51                        475.1     92.144      1.29
      380.8     98.307      0.63                        480.2     91.790      1.05
      385.2     98.154      0.56                        485.2     91.521      0.76
      390.3     97.981      0.57                        490.1     91.317      0.60
      395.6     97.804      0.66                        495.0     91.174      0.38
      400.7     97.598      0.68                        500.1     91.084      0.23
      405.0     97.425      0.71                        505.1     91.021      0.17
      410.2     97.198      0.82                        510.2     90.991      0.06
      415.5     96.948      0.89                        515.2     90.968      0.06
      420.7     96.662      0.97                        520.2     90.958      0.06
      425.0     96.415      1.03                        525.2     90.944      0.04
      430.2     96.082      1.16                        530.1     90.934      0.01
      435.3     95.722      1.26                        535.1     90.931      0.01
      440.4     95.326      1.39                        540.0     90.928      0.01
      445.6     94.896      1.48                        545.8     90.921      0.01
                                                        550.7     90.918      0.00
```

Sample: 90% SAND
Size: 26.76 mg
Rate: 20C/MIN
Program: TGA Analysis V2.0

TGA

Date: 30-Oct-85 Time: 13:18:23
File: PEL/RES.04 SCRAP BOOK JT
Operator: JT
Plotted: 1-Nov-85 11:35:04



```
Sample: 90% SAND                                      Date: 30-Oct-85    Time: 13:18:23
Size: 26.76 mg                                        File: PEL/RES.04 SCRAP BOOK JT
Rate: 20C/MIN                                         Operator: JT
Program: General Analysis V1.0                        Plotted:  1-Nov-85    11:47:51

     Temp        Sig-A      Deriv-A                      Temp       Sig-A      Deriv-A
     [deg]        [%]       [%/min]                      [deg]       [%]       [%/min]

     150.6      100.049       0.00                       250.5     100.060       0.03
     155.4      100.052      -0.02                       255.2     100.052       0.02
     160.0      100.056      -0.02                       260.0     100.052       0.03
     165.9      100.064      -0.01                       266.0     100.034       0.06
     170.6      100.064       0.00                       270.7     100.026       0.06
     175.4      100.060       0.01                       275.4     100.007       0.08
     180.1      100.060       0.00                       280.2      99.996       0.09
     186.0      100.056      -0.01                       286.1      99.963       0.10
     190.7      100.064      -0.02                       290.9      99.940       0.12
     195.5      100.067       0.00                       295.7      99.918       0.12
     200.3      100.064       0.02                       300.5      99.895       0.13
     205.0      100.064      -0.04                       305.3      99.865       0.19
     211.0      100.067      -0.02                       310.1      99.824       0.21
     215.8      100.078       0.00                       316.0      99.776       0.22
     220.6      100.071       0.00                       320.7      99.716       0.28
     225.5      100.075       0.02                       325.3      99.664       0.28
     230.2      100.064       0.00                       330.1      99.604       0.33
     235.0      100.067       0.00                       336.0      99.518       0.36
     241.0      100.064       0.02                       340.6      99.443       0.37
     245.7      100.064       0.01                       345.4      99.368       0.39
                                                         350.0      99.286       0.47
```

```
Sample: 90% SAND
Size: 26.76 mg                                          Date: 30-Oct-85    Time: 13:18:23
Rate: 20C/MIN                                           File: PEL/RES.04 SCRAP BOOK JT
Program: General Analysis V1.0                          Operator: JT
                                                        Plotted:  1-Nov-85    12:02:39

    Temp        Sig-A       Deriv-A                  Temp        Sig-A       Deriv-A
   [deg]         [%]        [%/min]                 [deg]         [%]        [%/min]

   350.0       99.286        0.47                   450.4       94.951        2.02
   355.9       99.167        0.49                   456.1       94.428        2.17
   360.6       99.058        0.53                   460.7       93.983        2.26
   365.3       98.954        0.56                   465.3       93.520        2.31
   370.0       98.838        0.58                   471.0       92.941        2.29
   376.0       98.681        0.61                   475.5       92.492        2.17
   380.7       98.565        0.64                   481.1       91.991        1.84
   385.4       98.427        0.73                   485.6       91.648        1.56
   390.1       98.270        0.79                   490.2       91.364        1.25
   396.0       98.068        0.85                   496.0       91.102        0.88
   400.7       97.900        0.89                   500.5       90.956        0.63
   405.4       97.713        0.97                   505.1       90.848        0.44
   410.0       97.511        1.03                   510.7       90.762        0.24
   415.8       97.250        1.11                   515.2       90.725        0.19
   420.5       97.010        1.21                   520.8       90.691        0.09
   425.2       96.767        1.28                   525.3       90.680        0.07
   430.9       96.431        1.46                   531.0       90.661        0.05
   435.6       96.117        1.58                   535.3       90.650        0.05
   440.2       95.792        1.73                   540.5       90.642        0.02
   445.9       95.340        1.88                   545.7       90.639        0.04
                                                    551.0       90.620        0.07
```

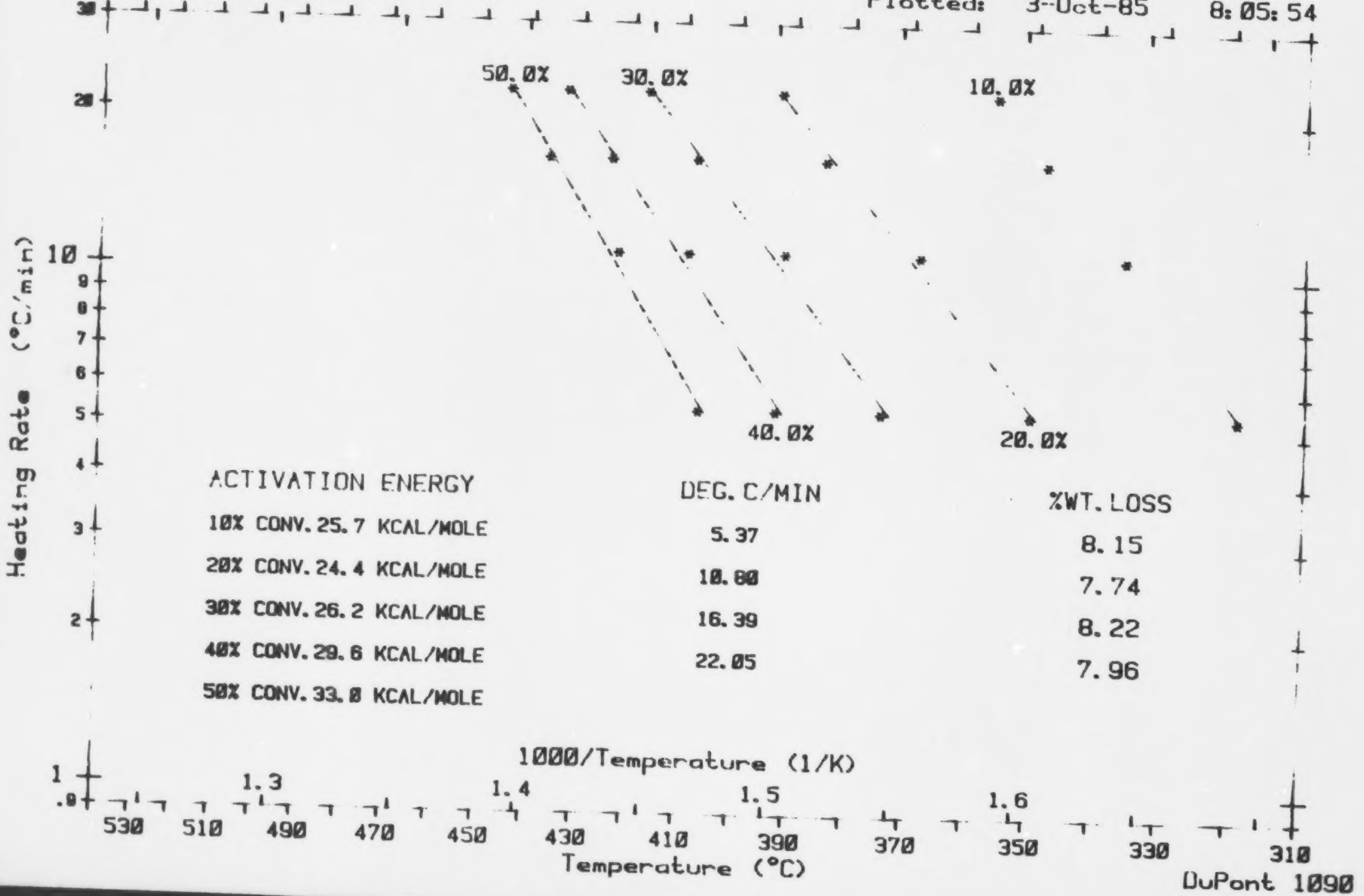
Sample: 10% KAOLIN

Date: 1-Oct-85 Time: 11:13:59

Program: TGA Kinetics V1.0

Operator: JT

Plotted: 3-Oct-85 8:05:54



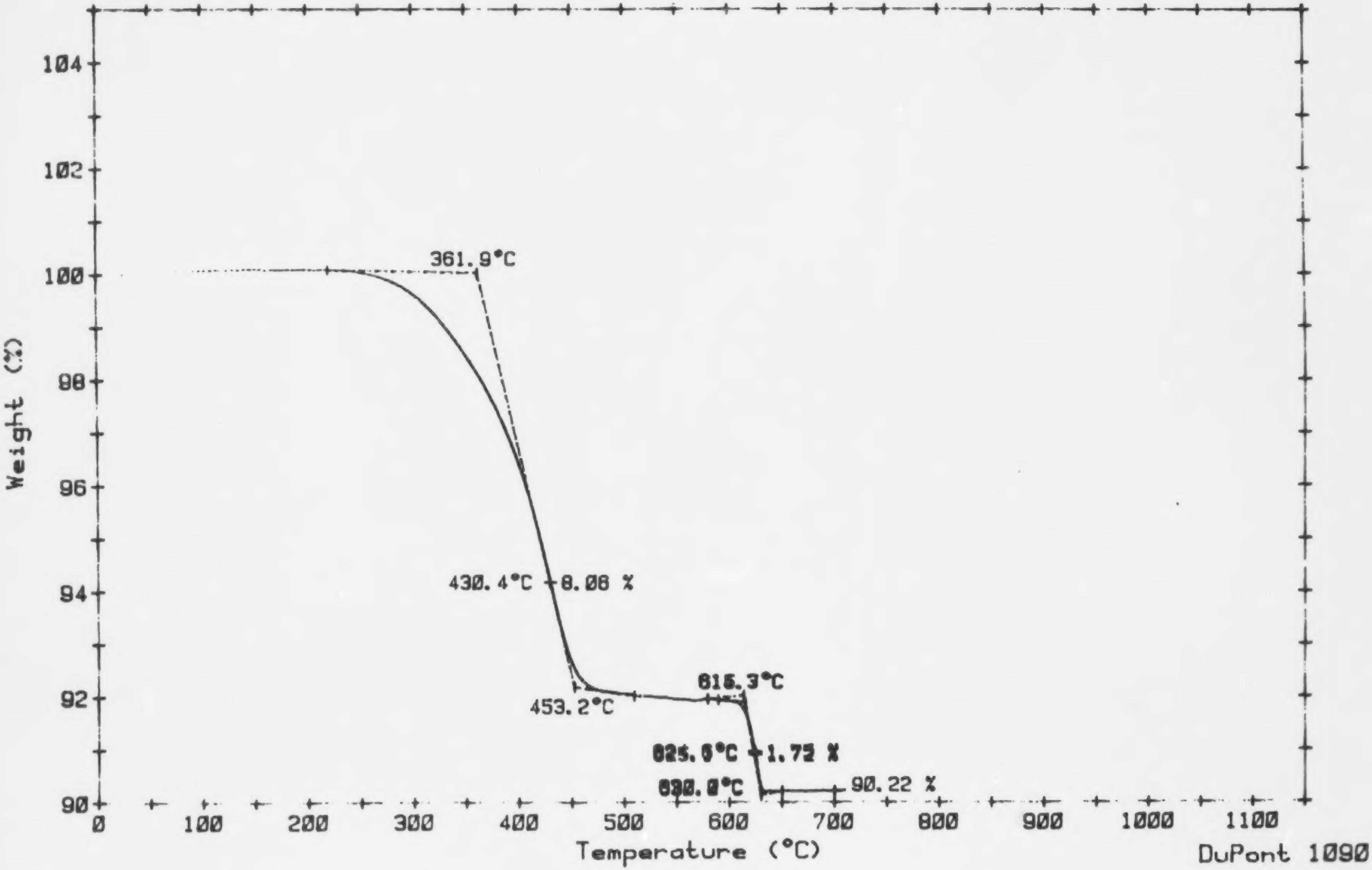
ACTIVATION ENERGY	DEG. C/MIN	%WT. LOSS
10% CONV. 25.7 KCAL/MOLE	5.37	8.15
20% CONV. 24.4 KCAL/MOLE	10.80	7.74
30% CONV. 26.2 KCAL/MOLE	16.39	8.22
40% CONV. 29.6 KCAL/MOLE	22.05	7.96
50% CONV. 33.0 KCAL/MOLE		

DuPont 1090

Sample: 10% KAOLIN
Size: 26.80 mg
Rate: 05C/MIN
Program: General Analysis V1.0

TGA

Date: 2-Oct-85 Time: 9:44:15
File: PEL/RESID.33 T.LYNCH.08
Operator: JT
Plotted: 2-Oct-85 11:45:14



DuPont 1090

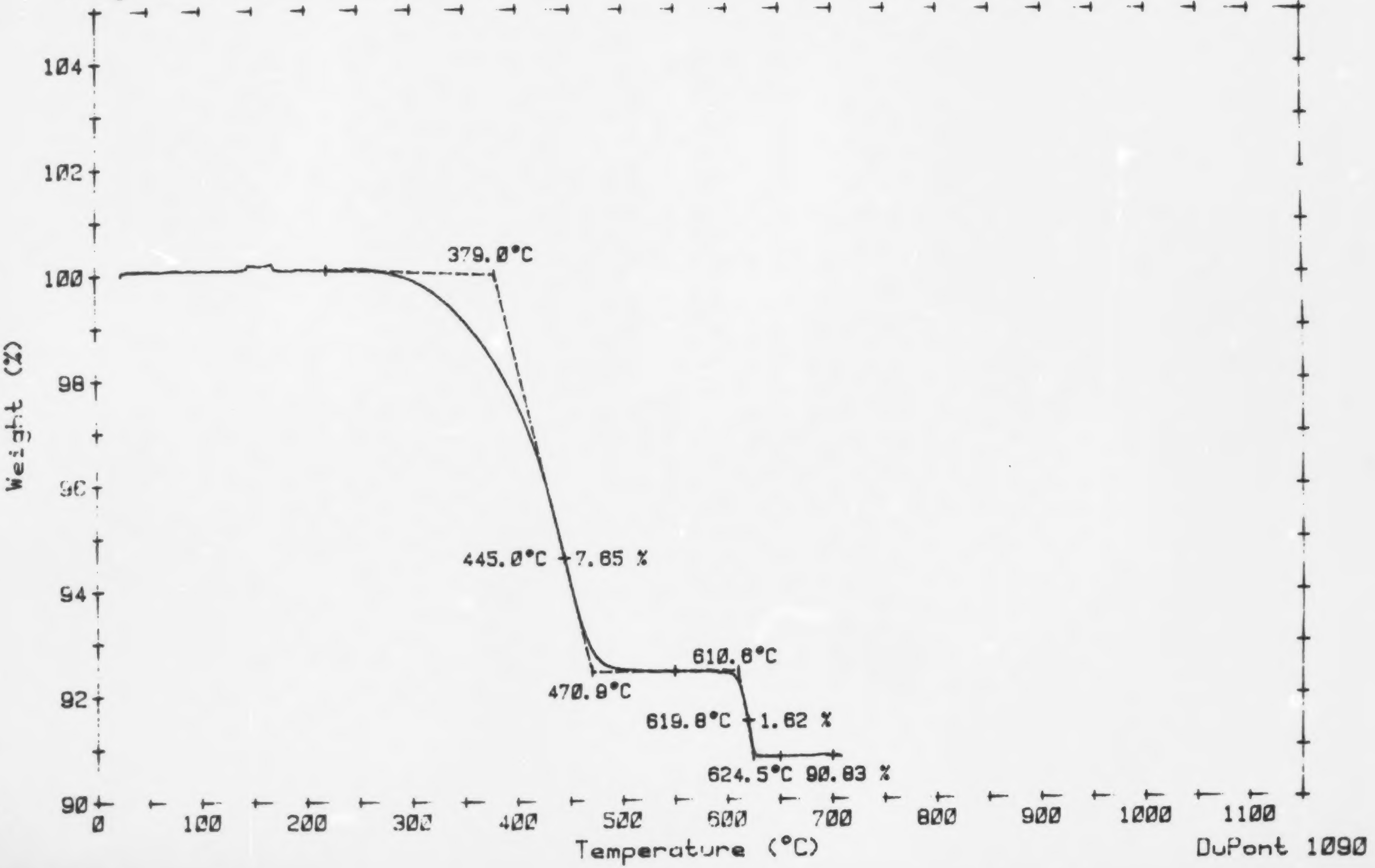
```
Sample: 10% KAOLIN                                      Date:  2-Oct-85   Time:  9:44:15
Size:  26.80 mg                                         File:  PEL/RESID.33 T.LYNCH.08
Rate:  05C/MIN                                          Operator:  JT
Program:  General Analysis V1.0                         Plotted:  3-Oct-85   11:01:36

     Temp       Sig-A      Deriv-A                    Temp       Sig-A      Deriv-A
     [deg]       [%]       [%/min]                    [deg]       [%]       [%/min]

     150.2     100.087       0.01                     250.2     100.026       0.00
     155.2     100.080       0.00                     255.2     100.011       0.01
     160.0     100.075       0.03                     260.2      99.989       0.03
     165.1     100.075      -0.02                     265.1      99.963       0.02
     170.0     100.078       0.00                     270.3      99.937       0.02
     175.1     100.071       0.00                     275.1      99.899       0.02
     180.3     100.078       0.01                     280.1      99.873       0.05
     185.3     100.082      -0.01                     285.1      99.817       0.08
     190.2     100.082       0.01                     290.2      99.765       0.05
     195.0     100.075      -0.02                     295.1      99.702       0.07
     200.2     100.082       0.00                     300.2      99.634       0.07
     205.2     100.075       0.00                     305.1      99.556       0.09
     210.1     100.078       0.00                     310.1      99.463       0.11
     215.2     100.071       0.02                     315.1      99.366       0.12
     220.2     100.075       0.00                     320.1      99.261       0.10
     225.2     100.078       0.01                     325.1      99.146       0.14
     230.1     100.067      -0.01                     330.0      99.030       0.16
     235.1     100.075       0.01                     335.2      98.892       0.15
     240.2     100.052       0.03                     340.1      98.761       0.16
     245.2     100.041       0.01                     345.1      98.619       0.17
                                                      350.1      98.470       0.17
```

```
Sample: 10% KAOLIN                                  Date:   2-Oct-85    Time:   9:44:15
Size:  26.80 mg                                     File:  PEL/RESID.33 T.LYNCH.08
Rate:  05C/MIN                                      Operator:  JT
Program:  General Analysis V1.0                     Plotted:   3-Oct-85    11:30:54

      Temp       Sig-A      Deriv-A                    Temp       Sig-A      Deriv-A
     [deg]        [%]       [%/min]                   [deg]        [%]       [%/min]

     350.1      98.470       0.17                     450.2      92.683       0.27
     355.2      98.314       0.18                     455.0      92.474       0.19
     360.1      98.161       0.17                     460.0      92.329       0.13
     365.0      97.996       0.18                     465.2      92.228       0.09
     370.0      97.817       0.20                     470.1      92.176       0.04
     375.0      97.616       0.21                     475.0      92.139       0.03
     380.0      97.429       0.23                     480.2      92.116       0.01
     385.1      97.194       0.23                     485.1      92.105       0.03
     390.1      96.959       0.24                     490.1      92.090       0.03
     395.0      96.717       0.27                     495.1      92.064       0.02
     400.0      96.437       0.29                     500.1      92.053       0.00
     405.1      96.135       0.34                     505.2      92.049       0.00
     410.1      95.799       0.35                     510.2      92.019       0.00
     415.1      95.433       0.41                     515.2      92.019      -0.01
     420.2      95.041       0.42                     520.0      92.012       0.00
     425.1      94.657       0.44                     525.0      92.004       0.01
     430.1      94.198       0.46                     530.2      91.986       0.01
     435.1      93.765       0.45                     535.0      91.989       0.02
     440.1      93.355       0.43                     540.2      91.986       0.00
     445.2      92.974       0.35                     545.1      91.971       0.03
                                                      550.0      91.948       0.00
```

Sample: 10% KAOLIN
Size: 26.55 mg
Rate: 10C/MIN
Program: General Analysis V1.0

TGA

Date: 2-Oct-85 Time: 15:37:37
File: PEL/RESID.35 T.LYNCH.08
Operator: JT
Plotted: 3-Oct-85 7:51:28



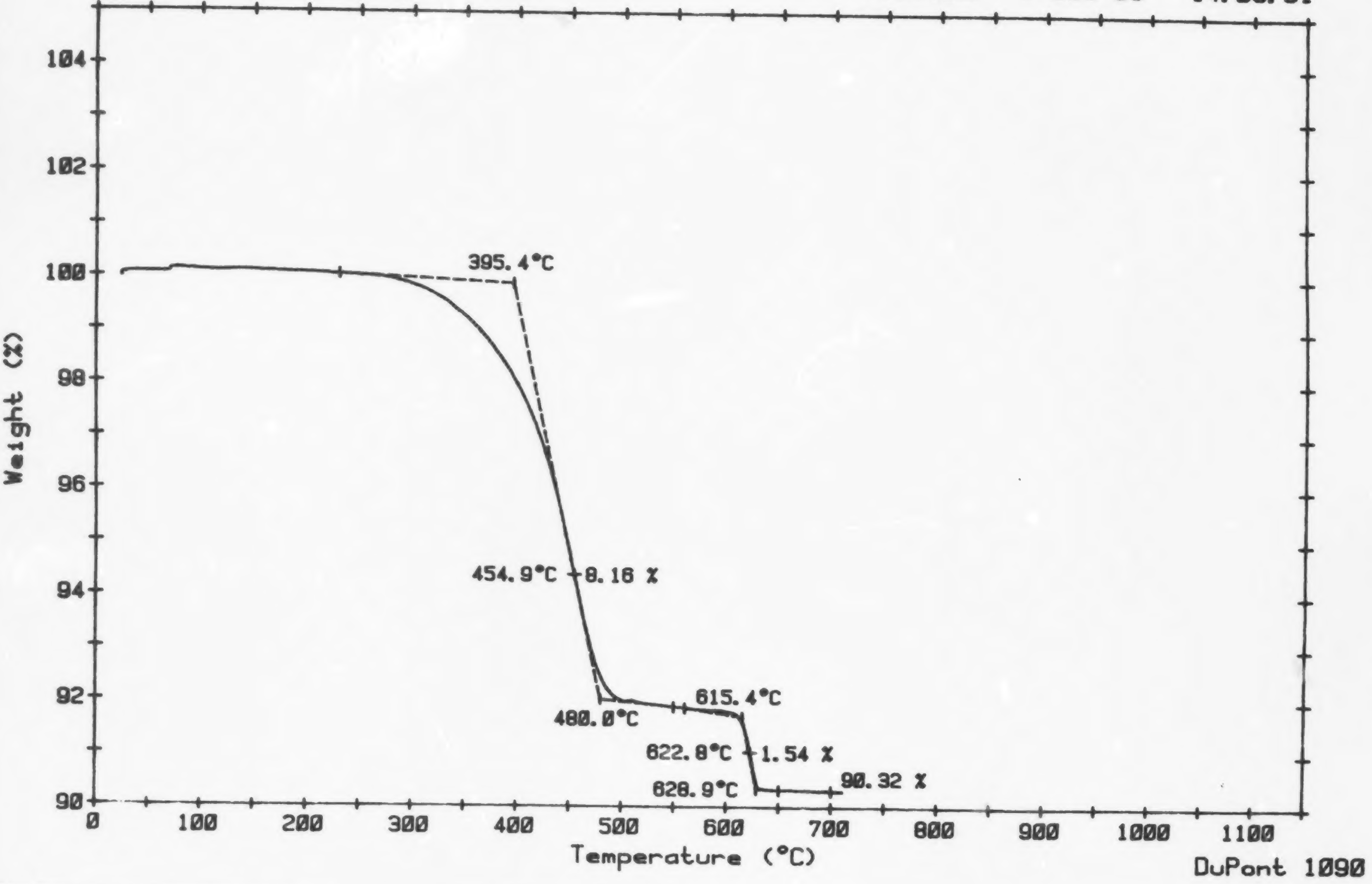
```
Sample: 10% KAOLIN                                      Date:   2-Oct-85   Time: 15:37:37
Size:  26.55 mg                                         File:  PEL/RESID.35 T.LYNCH.08
Rate:  10C/MIN                                          Operator:  JT
Program:  General Analysis V1.0                         Plotted:   3-Oct-85    11:41:21

      Temp        Sig-A       Deriv-A                        Temp        Sig-A       Deriv-A
     [deg]         [%]        [%/min]                       [deg]         [%]        [%/min]

     150.4       100.170        0.05                        250.5       100.098        0.01
     155.0       100.151        0.02                        255.0       100.087        0.04
     160.2       100.158       -0.01                        260.0       100.079        0.03
     165.3       100.181       -0.05                        265.4       100.060        0.02
     170.5       100.185        0.40                        270.3       100.041        0.03
     175.5       100.072       -0.01                        275.4       100.026        0.04
     180.0       100.072        0.00                        280.3       100.000        0.07
     185.2       100.072        0.02                        285.1        99.981        0.05
     190.4       100.079       -0.08                        290.1        99.955        0.07
     195.0       100.087        0.00                        295.1        99.921        0.08
     200.0       100.087        0.02                        300.1        99.883        0.09
     205.3       100.079        0.01                        305.0        99.838        0.12
     210.4       100.079       -0.01                        310.5        99.778        0.12
     215.3       100.075       -0.02                        315.5        99.710        0.14
     220.4       100.075        0.01                        320.4        99.650        0.14
     225.5       100.072        0.02                        325.3        99.574        0.18
     230.5       100.068        0.02                        330.2        99.495        0.20
     235.5       100.056       -0.08                        335.2        99.405        0.20
     240.4       100.098       -0.01                        340.0        99.307        0.21
     245.4       100.102       -0.01                        345.4        99.190        0.24
                                                            350.3        99.085        0.24
```

```
Sample: 10% KAOLIN                                          Date:   2-Oct-85    Time: 15:37:37
Size: 26.55 mg                                              File: PEL/RESID.35 T.LYNCH.08
Rate: 10C/MIN                                               Operator: JT
Program: General Analysis V1.0                              Plotted:   3-Oct-85    11:53:32

      Temp        Sig-A       Deriv-A                  Temp        Sig-A       Deriv-A
      [deg]        [%]        [%/min]                  [deg]        [%]        [%/min]

      350.3       99.085       0.24                    450.3       94.146       0.89
      355.2       98.964       0.27                    455.5       93.728       0.79
      360.1       98.840       0.27                    460.2       93.389       0.69
      365.0       98.716       0.30                    465.5       93.084       0.55
      370.5       98.561       0.32                    470.2       92.866       0.40
      375.3       98.410       0.34                    475.4       92.700       0.25
      380.1       98.260       0.36                    480.0       92.606       0.17
      385.0       98.086       0.39                    485.2       92.542       0.10
      390.3       97.891       0.40                    490.4       92.504       0.08
      395.1       97.714       0.41                    495.1       92.482       0.04
      400.4       97.488       0.46                    500.2       92.466       0.02
      405.3       97.269       0.52                    505.3       92.459       0.02
      410.0       97.032       0.56                    510.4       92.455      -0.01
      415.3       96.742       0.60                    515.5       92.440       0.03
      420.1       96.459       0.68                    520.0       92.436       0.00
      425.4       96.109       0.71                    525.5       92.433       0.01
      430.2       95.770       0.79                    530.0       92.425       0.02
      435.5       95.363       0.84                    535.0       92.421       0.01
      440.2       94.975       0.87                    540.0       92.425      -0.01
      445.0       94.580       0.87                    545.1       92.421       0.01
                                                       550.1       92.425       0.00
```

Sample: 10% KAOLIN
Size: 27.81 mg
Rate: 15C/MIN
Program: General Analysis V1.0

TGA

Date: 1-Oct-85 Time: 13:11:22
File: PEL/RESID.31 T.LYNCH.08
Operator: JT
Plotted: 1-Oct-85 14:08:51



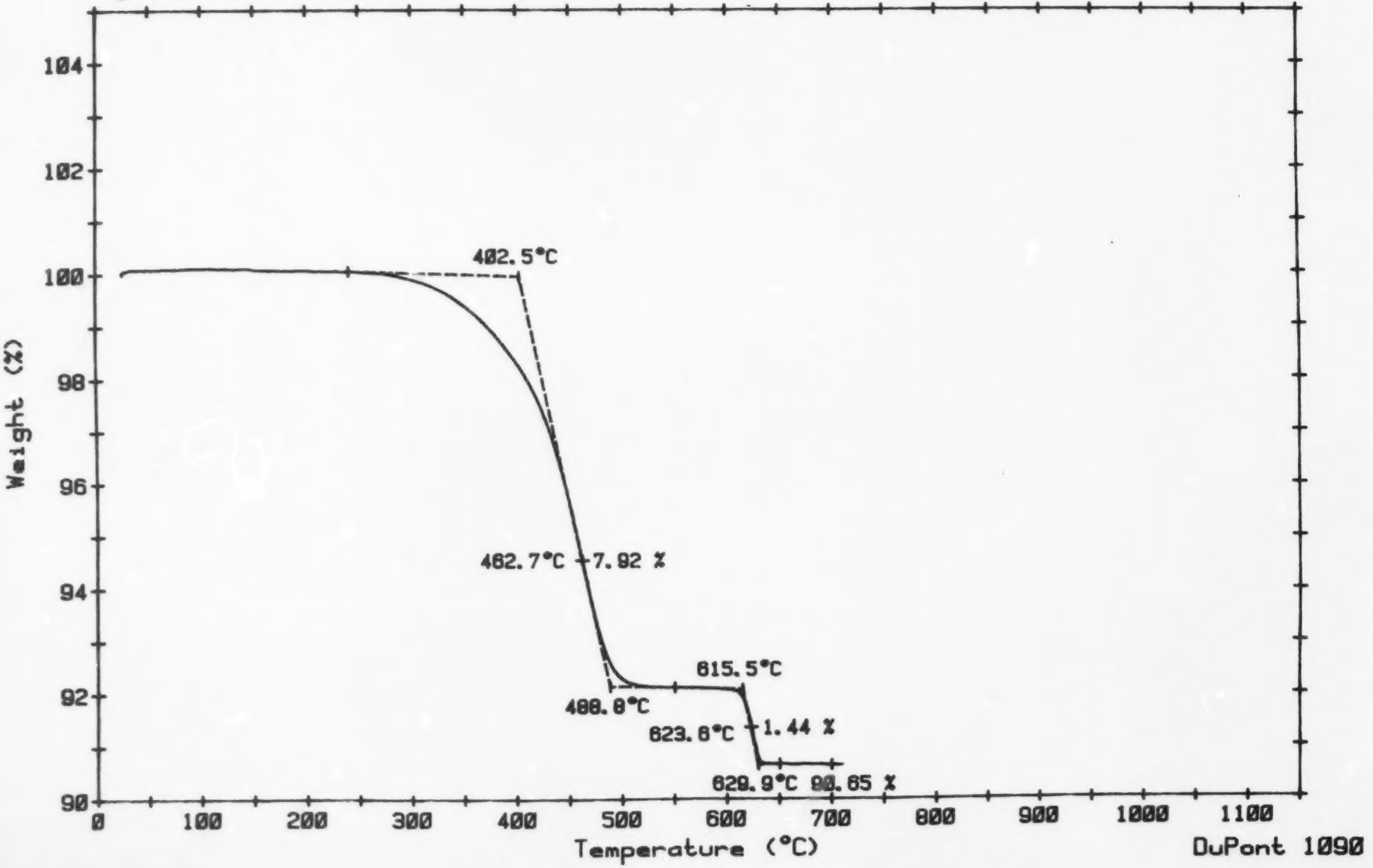
```
Sample: 10% KAOLIN                                    Date:  1-Oct-85   Time: 13:11:22
Size: 27.81 mg                                        File: PEL/RESID.31 T.LYNCH.08
Rate: 15C/MIN                                         Operator: JT
Program: General Analysis V1.0                        Plotted:  3-Oct-85   12:46:15

     Temp       Sig-A      Deriv-A                        Temp      Sig-A      Deriv-A
     [deg]       [%]       [%/min]                       [deg]       [%]       [%/min]

     150.8     100.115       0.02                        250.2     100.029       0.01
     155.3     100.108       0.03                        255.3     100.025       0.02
     160.5     100.108      -0.01                        260.3     100.018       0.02
     165.7     100.108       0.02                        265.5     100.011       0.03
     170.1     100.101       0.03                        270.5     100.007       0.05
     175.3     100.093       0.00                        275.6      99.986       0.05
     180.5     100.093       0.01                        280.6      99.971       0.07
     185.6     100.086       0.00                        285.6      99.942       0.08
     190.8     100.083       0.02                        290.6      99.921       0.08
     195.1     100.079       0.02                        295.6      99.896       0.09
     200.2     100.079       0.00                        300.7      99.860       0.12
     205.3     100.076       0.02                        305.7      99.827       0.13
     210.3     100.072       0.02                        310.7      99.784       0.15
     215.6     100.065       0.01                        315.7      99.734       0.17
     220.8     100.061       0.03                        320.7      99.687       0.18
     225.0     100.054       0.02                        325.7      99.630       0.18
     230.8     100.054       0.00                        330.7      99.572       0.22
     235.0     100.047       0.03                        335.5      99.496       0.28
     240.0     100.036       0.03                        340.5      99.417       0.27
     245.2     100.036       0.01                        345.4      99.338       0.26
                                                         350.5      99.259       0.28
```

```
Sample: 10% KAOLIN                                          Date:  1-Oct-85   Time: 13:11:22
Size: 27.81 mg                                              File: PEL/RESID.31 T.LYNCH.08
Rate: 15C/MIN                                               Operator: JT
Program: General Analysis V1.0                              Plotted:  3-Oct-85   13:01:54

      Temp       Sig-A      Deriv-A                           Temp       Sig-A      Deriv-A
      [deg]      [%]        [%/min]                           [deg]      [%]        [%/min]

      350.5      99.259       0.28                            450.2      94.839       1.49
      355.4      99.169       0.31                            455.7      94.310       1.49
      360.3      99.069       0.36                            460.4      93.868       1.48
      365.3      98.961       0.38                            465.2      93.440       1.33
      370.2      98.849       0.38                            470.7      93.005       1.14
      375.1      98.723       0.43                            475.5      92.692       0.91
      380.8      98.572       0.45                            480.2      92.451       0.70
      385.7      98.436       0.47                            485.7      92.253       0.45
      390.6      98.292       0.52                            490.4      92.134       0.32
      395.4      98.123       0.58                            495.1      92.063       0.20
      400.3      97.950       0.59                            500.6      92.012       0.12
      405.2      97.763       0.68                            505.2      91.983       0.01
      410.1      97.554       0.72                            510.7      92.012       0.04
      415.8      97.285       0.82                            515.2      91.976       0.07
      420.6      97.033       0.88                            520.5      91.958       0.05
      425.3      96.749       0.96                            525.1      91.944       0.03
      430.2      96.447       1.07                            530.4      91.930       0.04
      435.0      96.105       1.16                            535.0      91.922       0.02
      440.6      95.681       1.31                            540.4      91.919       0.04
      445.4      95.267       1.41                            545.6      91.908       0.03
                                                              550.1      91.897       0.04
```

Sample: 10% KAOLIN
Size: 26.67 mg
Rate: 20C/MIN
Program: General Analysis V1.0

TGA

Date: 1-Oct-85 Time: 11:13:59
File: PEL/RESID.30 T.LYNCH.08
Operator: JT
Plotted: 1-Oct-85 12:16:33



DuPont 1090

```
Sample: 10% KAOLIN                                      Date:  1-Oct-85   Time: 11:13:59
Size: 26.67 mg                                          File: PEL/RESID.30 T.LYNCH.08
Rate: 20C/MIN                                           Operator: JT
Program: General Analysis V1.0                          Plotted:  3-Oct-85   13:48:00

     Temp      Sig-A      Deriv-A                            Temp      Sig-A      Deriv-A
     [deg]      [%]       [%/min]                            [deg]      [%]       [%/min]

     150.5    100.075       0.09                             251.0    100.026       0.02
     155.3    100.067       0.02                             255.5    100.022       0.05
     160.0    100.064       0.02                             260.1    100.007       0.03
     166.0    100.064      -0.01                             265.6    100.011       0.03
     170.7    100.064       0.01                             270.1    100.000       0.05
     175.6    100.060       0.01                             275.7     99.985       0.06
     180.3    100.060       0.00                             280.2     99.974       0.09
     185.1    100.060       0.01                             285.9     99.947       0.10
     191.0    100.064      -0.01                             290.3     99.925       0.11
     195.7    100.064       0.01                             295.9     99.899       0.10
     200.4    100.060       0.03                             300.4     99.876       0.12
     205.1    100.052       0.02                             306.0     99.846       0.14
     210.9    100.058      -0.01                             310.5     99.816       0.16
     215.6    100.052       0.02                             315.0     99.782       0.17
     220.2    100.045       0.02                             320.6     99.741       0.21
     225.9    100.049       0.00                             325.0     99.696       0.23
     230.4    100.045       0.04                             330.7     99.636       0.24
     235.0    100.037       0.02                             335.1     99.587       0.27
     240.8    100.037       0.00                             340.6     99.512       0.31
     245.3    100.034       0.03                             345.0     99.449       0.31
                                                             350.6     99.370       0.34
```

```
Sample: 10% KAOLIN                                     Date:   1-Oct-85    Time: 11:13:59
Size:  26.67 mg                                        File:  PEL/RESID.30 T.LYNCH.08
Rate:  20C/MIN                                         Operator:  JT
Program:  General Analysis V1.0                        Plotted:   3-Oct-85    13:56:02

      Temp        Sig-A       Deriv-A                        Temp        Sig-A       Deriv-A
     [deg]         [%]        [%/min]                       [deg]         [%]        [%/min]

     350.6       99.370         0.34                        450.0       95.657         1.74
     355.1       99.302         0.37                        455.3       95.200         1.89
     360.6       99.205         0.41                        460.6       94.720         1.95
     365.1       99.119         0.43                        465.9       94.232         1.95
     370.6       99.010         0.47                        470.1       93.842         1.88
     375.0       98.912         0.50                        475.4       93.396         1.67
     380.5       98.785         0.53                        480.7       93.014         1.39
     386.0       98.654         0.55                        486.0       92.706         1.05
     390.3       98.541         0.56                        490.2       92.526         0.76
     395.8       98.395         0.61                        495.5       92.372         0.50
     400.2       98.271         0.64                        500.6       92.275         0.31
     405.7       98.106         0.72                        505.8       92.211         0.19
     410.1       97.952         0.79                        510.9       92.181         0.11
     415.6       97.746         0.87                        515.0       92.159         0.11
     421.0       97.517         1.00                        520.1       92.140         0.04
     425.3       97.300         1.09                        525.2       92.136         0.03
     430.7       97.026         1.22                        530.3       92.125         0.00
     435.0       96.760         1.41                        535.4       92.125         0.02
     440.4       96.389         1.52                        540.4       92.121         0.02
     445.8       95.995         1.65                        545.5       92.114         0.00
                                                            550.6       92.114         0.01
```

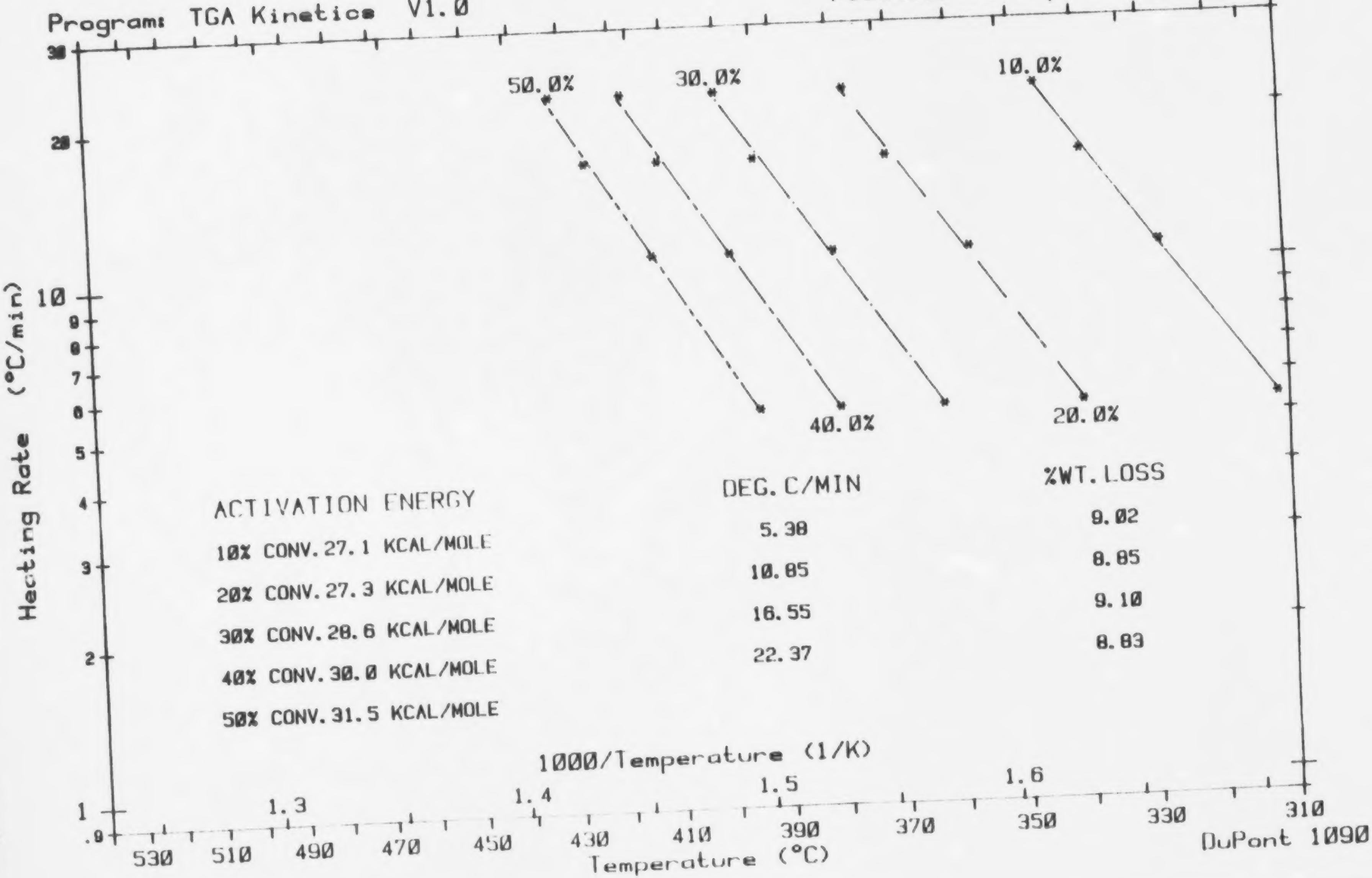
Sample: KAOLIN 45%

Date: 18-Sep-85 Time: 12:23:56

Operator: JT

Plotted: 26-Sep-85 14:36:49

Program: TGA Kinetics V1.0



ACTIVATION ENERGY

10% CONV. 27.1 KCAL/MOLE

20% CONV. 27.3 KCAL/MOLE

30% CONV. 28.6 KCAL/MOLE

40% CONV. 30.0 KCAL/MOLE

50% CONV. 31.5 KCAL/MOLE

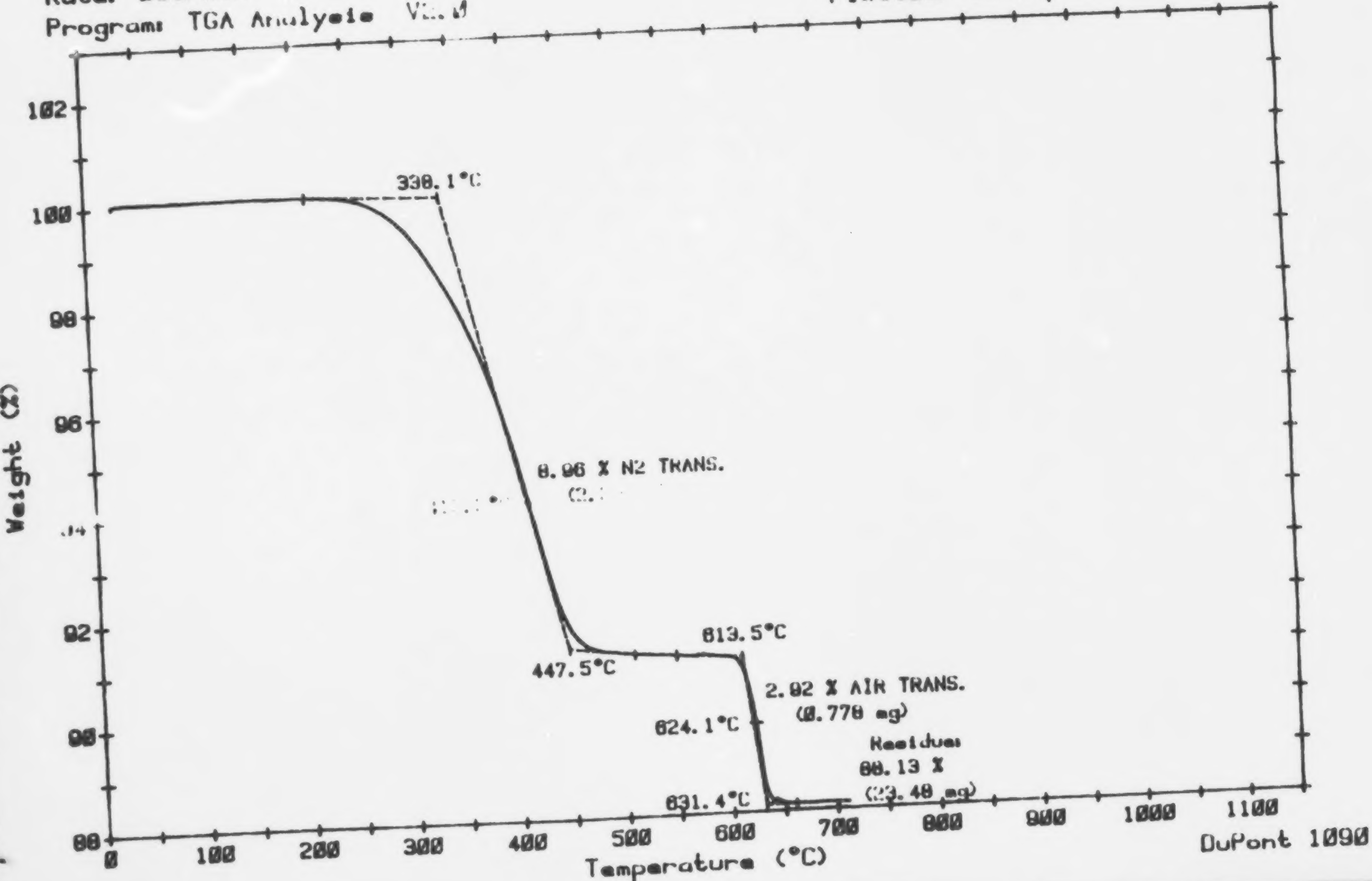
DEG. C/MIN	%WT. LOSS
5.38	9.02
10.85	8.85
16.55	9.10
22.37	8.83

DuPont 1090

Sample: KAOLIN 45%
Size: 26.64 mg
Rate: 05C/MIN
Program: TGA Analysis V[illegible]

TGA

Date: 19-Sep-85 Time: 13:10:15
File: PEL-RESID.09 T.LYNCH.07
Operator: JT
Plotted: 19-Sep-85 15:24:38



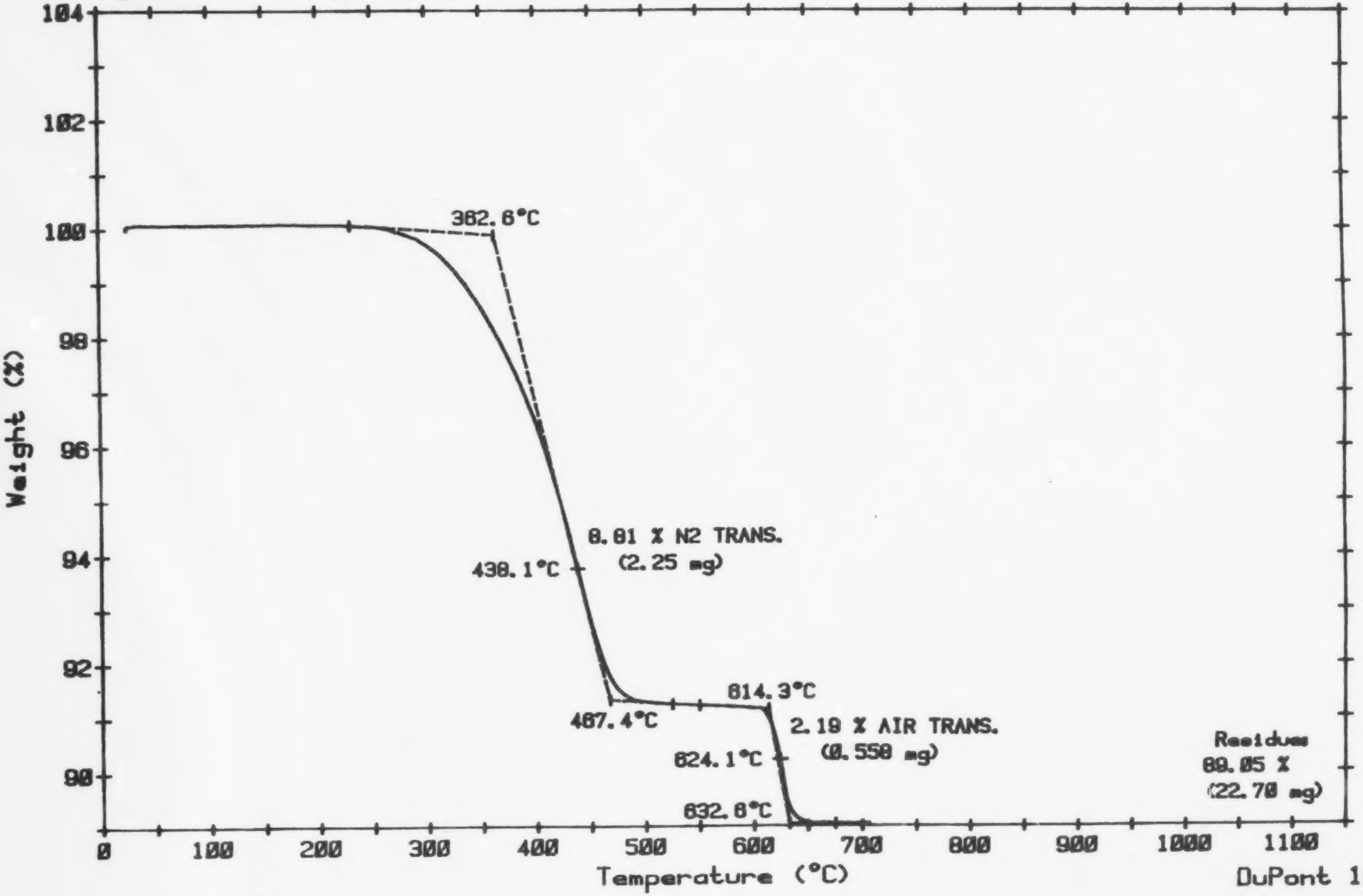
```
Sample: KAOLIN 45%                                  Date: 19-Sep-85    Time: 13:10:15
Size: 26.64 mg                                      File: PEL-RESID.81 T.LYNCH.07
Rate: 05C/MIN                                       Operator: JT
Program: General Analysis V1.0                      Plotted: 23-Sep-85    16:28:35

    Temp       Sig-A       Deriv-A                     Temp       Sig-A      Deriv-A
    [deg]       [%]        [%/min]                     [deg]       [%]       [%/min]

    150.1     100.079        0.00                      250.2     99.996        0.01
    155.1     100.079        0.00                      255.2     99.974        0.01
    160.2     100.083        0.00                      260.1     99.951        0.02
    165.3     100.083        0.00                      265.1     99.914        0.02
    170.0     100.079        0.00                      270.0     99.872        0.03
    175.1     100.079        0.00                      275.2     99.820        0.03
    180.2     100.079        0.01                      280.1     99.764        0.03
    185.2     100.075        0.00                      285.0     99.703        0.04
    190.1     100.079        0.00                      290.0     99.625        0.05
    195.2     100.079       -0.01                      295.2     99.535        0.05
    200.0     100.068        0.00                      300.1     99.433        0.06
    205.0     100.068        0.00                      305.1     99.336        0.06
    210.1     100.064        0.02                      310.2     99.208        0.07
    215.2     100.060        0.01                      315.2     99.073        0.08
    220.0     100.060        0.00                      320.2     98.934        0.09
    225.2     100.060        0.00                      325.0     98.780        0.10
    230.1     100.045        0.01                      330.0     98.622        0.09
    235.2     100.038        0.01                      335.2     98.438        0.10
    240.0     100.030        0.01                      340.2     98.270        0.11
    245.0     100.015        0.01                      345.0     98.089        0.11
                                                       350.2     97.890        0.11
```

```
Sample: KAOLIN 45%                                  Date: 19-Sep-85    Time: 13:10:15
Size: 26.64 mg                                      File: PEL-RESID.81 T.LYNCH.07
Rate: 05C/MIN                                       Operator: JT
Program: General Analysis V1.0                      Plotted: 23-Sep-85    16:54:24

      Temp       Sig-A      Deriv-A                      Temp       Sig-A      Deriv-A
      [deg]      [%]        [%/min]                      [deg]      [%]        [%/min]

      350.2      97.890       0.11                       450.2      91.592       0.09
      355.1      97.680       0.12                       455.1      91.453       0.07
      360.1      97.459       0.13                       460.1      91.355       0.05
      365.1      97.226       0.13                       465.2      91.280       0.02
      370.2      96.971       0.14                       470.0      91.238       0.03
      375.0      96.715       0.15                       475.2      91.209       0.02
      380.1      96.434       0.15                       480.2      91.179       0.01
      385.0      96.145       0.17                       485.0      91.167       0.00
      390.1      95.815       0.18                       490.1      91.156       0.02
      395.2      95.469       0.19                       495.2      91.134       0.01
      400.1      95.131       0.19                       500.2      91.126       0.01
      405.1      94.748       0.21                       505.0      91.122       0.00
      410.1      94.343       0.22                       510.0      91.107       0.00
      415.1      93.928       0.20                       515.2      91.098       0.00
      420.1      93.532       0.22                       520.0      91.089       0.00
      425.0      93.123       0.22                       525.0      91.085       0.00
      430.2      92.714       0.20                       530.0      91.074       0.00
      435.1      92.361       0.17                       535.1      91.074       0.01
      440.2      92.042       0.14                       540.2      91.062       0.00
      445.1      91.798       0.12                       545.2      91.059       0.01
                                                         550.1      91.047       0.00
```

Sample: KAOLIN 45%
Size: 25.49 mg
Rate: 10C/MIN
Program: TGA Analysis V2.0

TGA

Date: 18-Sep-85 Time: 8:33:00
File: PEL-RESID.85 T.LYNCH.07
Operator: JT
Plotted: 18-Sep-85 9:51:28



DuPont 1090

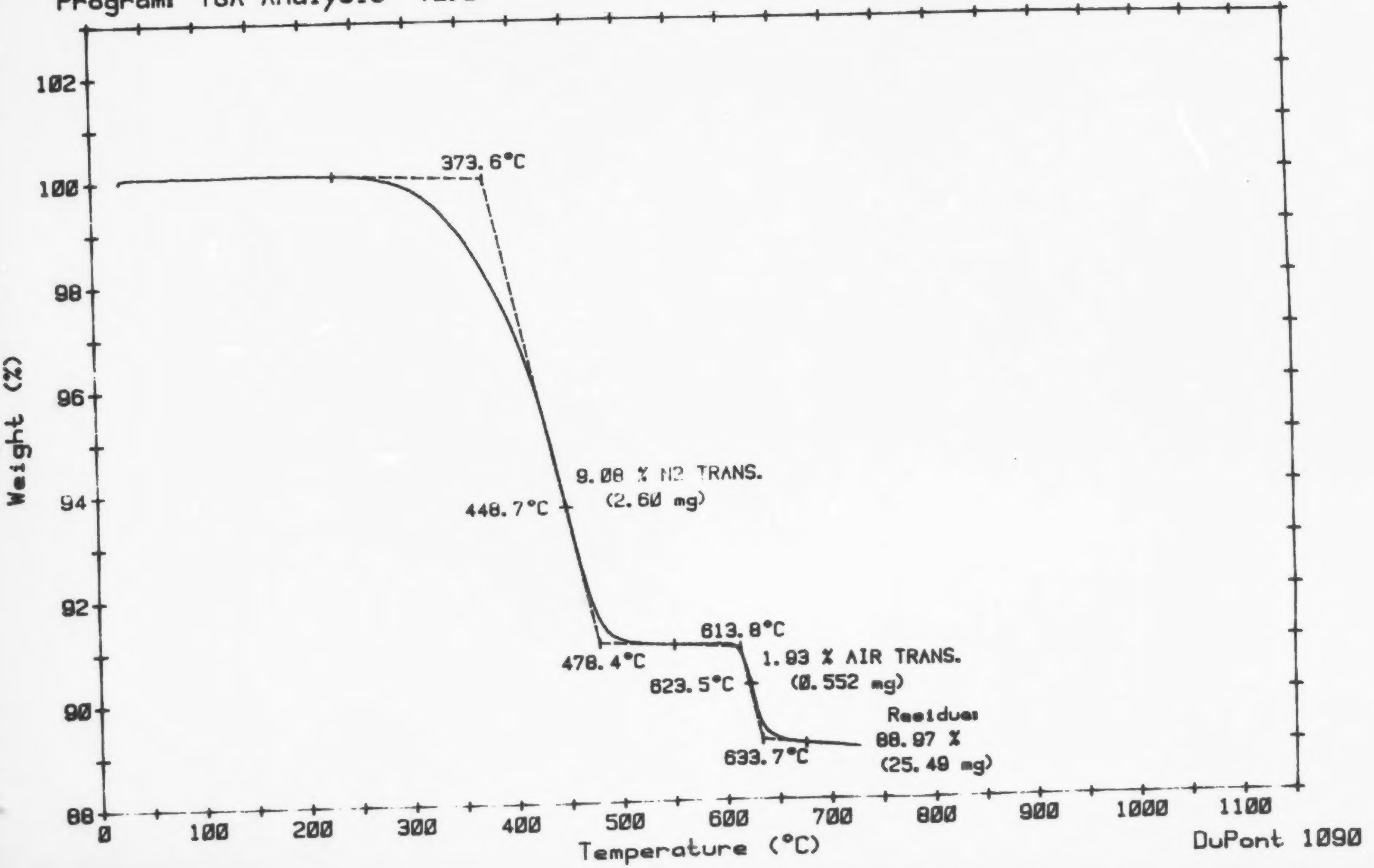
```
Sample: KAOLIN 45%                                    Date: 18-Sep-85    Time:  8:33:00
Size: 25.49 mg                                        File: PEL-RESID.82 T.LYNCH.07
Rate: 10C/MIN                                         Operator: JT
Program: General Analysis V1.0                        Plotted: 24-Sep-85   10:47:13

      Temp       Sig-A      Deriv-A                 Temp      Sig-A      Deriv-A
      [deg]       [%]       [%/min]                 [deg]      [%]       [%/min]

      150.0     100.086      -0.02                  250.3    100.051       0.03
      155.4     100.090      -0.01                  255.4    100.035       0.02
      160.1     100.090       0.00                  260.5    100.020       0.03
      165.4     100.090       0.01                  265.0    100.008       0.05
      170.0     100.086      -0.01                  270.1     99.965       0.06
      175.3     100.090       0.02                  275.1     99.937       0.04
      180.0     100.086       0.00                  280.1     99.902       0.10
      185.3     100.090       0.00                  285.1     99.871       0.08
      190.0     100.090       0.00                  290.1     99.839       0.10
      195.2     100.082       0.00                  295.0     99.776       0.14
      200.5     100.086       0.02                  300.0     99.718       0.14
      205.1     100.078       0.00                  305.0     99.655       0.17
      210.3     100.082       0.01                  310.4     99.565       0.19
      215.5     100.082       0.00                  315.4     99.474       0.22
      220.2     100.078      -0.01                  320.3     99.368       0.24
      225.3     100.078       0.01                  325.2     99.259       0.25
      230.4     100.071       0.01                  330.1     99.137       0.27
      235.5     100.067       0.02                  335.1     99.011       0.29
      240.0     100.055       0.00                  340.5     98.854       0.31
      245.2     100.055       0.01                  345.4     98.713       0.34
                                                    350.3     98.556       0.37
```

```
Sample: KAOLIN 45%                                    Date: 18-Sep-85    Time:   8:33:00
Size: 25.49 mg                                        File: PEL-RESID.82 T.LYNCH.07
Rate: 10C/MIN                                         Operator: JT
Program: General Analysis V1.0                        Plotted: 24-Sep-85    11:18:19

     Temp       Sig-A      Deriv-A                      Temp       Sig-A      Deriv-A
     [deg]      [%]        [%/min]                      [deg]      [%]        [%/min]

     350.3      98.556      0.37                        450.3      92.774      0.80
     355.2      98.392      0.37                        455.1      92.437      0.71
     360.1      98.219      0.39                        460.3      92.107      0.60
     365.5      98.023      0.40                        465.1      91.872      0.47
     370.3      97.835      0.44                        470.3      91.668      0.32
     375.2      97.638      0.45                        475.4      91.522      0.22
     380.0      97.427      0.48                        480.2      91.436      0.17
     385.4      97.191      0.51                        485.3      91.373      0.10
     390.2      96.960      0.53                        490.5      91.334      0.06
     395.0      96.713      0.57                        495.1      91.318      0.02
     400.4      96.430      0.60                        500.2      91.299      0.02
     405.2      96.156      0.64                        505.4      91.287      0.03
     410.0      95.857      0.68                        510.4      91.275      0.00
     415.3      95.504      0.73                        515.0      91.271      0.01
     420.1      95.163      [illegible]                 520.0      91.264      0.01
     425.4      94.775      0.81                        525.1      91.260      0.02
     430.3      94.398      0.85                        530.1      91.252      0.00
     435.5      93.966      0.88                        535.0      91.244      0.00
     440.2      93.574      0.88                        540.5      91.236      0.01
     445.0      93.182      0.86                        545.4      91.232      0.02
                                                        550.4      91.236      0.03
```

Sample: KAOLIN 45%
Size: 28.65 mg
Rate: 15C/MIN
Program: TGA Analysis V2.0

TGA

Date: 17-Sep-85 Time: 10:03:24
File: PEL-RESID.80 T.LYNCH.07
Operator: JT
Plotted: 17-Sep-85 11:08:19



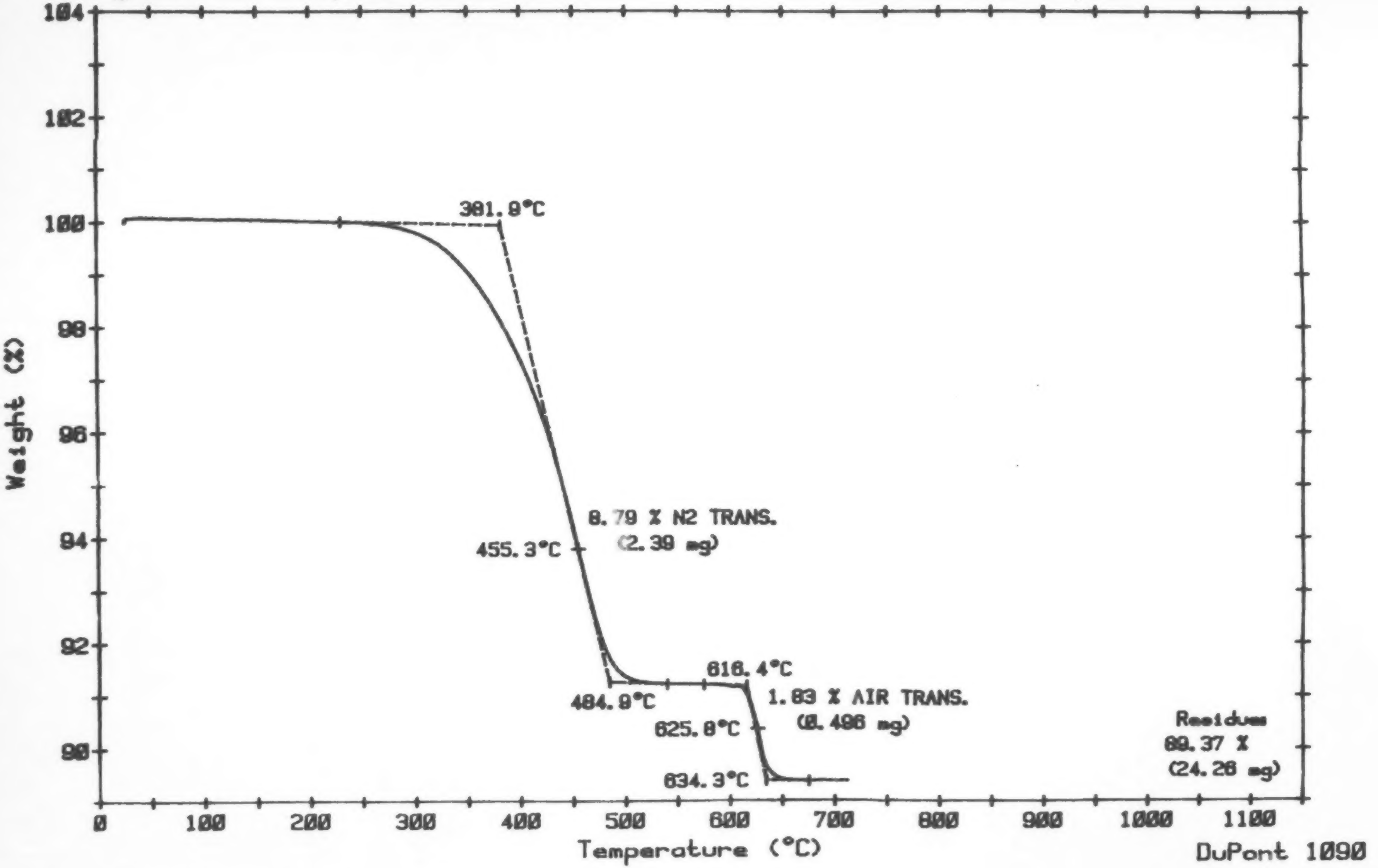
```
Sample: KAOLIN 45%                                  Date: 17-Sep-85   Time: 10:03:24
Size: 28.65 mg                                      File: PEL-RESID.83 T.LYNCH.07
Rate: 15C/MIN                                       Operator: JT
Program: General Analysis V1.0                      Plotted: 24-Sep-85   15:47:12

     Temp       Sig-A      Deriv-A                     Temp       Sig-A      Deriv-A
     [deg]       [%]       [%/min]                     [deg]       [%]       [%/min]

     150.7     100.098       0.01                      250.6     100.056       0.02
     155.2     100.098       0.00                      255.7     100.052       0.03
     160.6     100.098       0.00                      260.0     100.042       0.04
     165.0     100.098      -0.01                      265.1     100.031       0.04
     170.3     100.101       0.01                      270.3     100.014       0.09
     175.6     100.094       0.01                      275.4      99.986       0.09
     180.0     100.094       0.00                      280.4      99.962       0.06
     185.2     100.098       0.01                      285.4      99.937       0.10
     190.5     100.098       0.00                      290.5      99.906       0.11
     195.8     100.098       0.02                      295.6      99.867       0.14
     200.1     100.091       0.00                      300.6      99.826       0.15
     205.4     100.091       0.01                      305.7      99.777       0.20
     210.6     100.094       0.00                      310.7      99.710       0.21
     215.0     100.087       0.02                      315.0      99.654       0.25
     220.1     100.084       0.01                      320.2      99.571       0.28
     225.3     100.080       0.00                      325.3      99.487       0.30
     230.6     100.080       0.01                      330.3      99.389       0.35
     235.0     100.080       0.03                      335.2      99.281       0.38
     240.2     100.073       0.01                      340.3      99.162       0.41
     245.4     100.066       0.03                      345.2      99.037       0.37
                                                       350.2      98.922       0.44
```

```
Sample: KAOLIN 45%                                   Date: 17-Sep-85    Time: 10:03:24
Size: 28.65 mg                                       File: PEL-RESID.83 T.LYNCH.07
Rate: 15C/MIN                                        Operator: JT
Program: General Analysis V1.0                       Plotted: 24-Sep-85    15:55:26

     Temp       Sig-A      Deriv-A                    Temp       Sig-A      Deriv-A
     [deg]      [%]        [%/min]                    [deg]      [%]        [%/min]

     350.2      98.922      0.44                      450.3      93.516      1.36
     355.1      98.789      0.47                      455.0      93.107      1.36
     360.7      98.608      0.53                      460.5      92.647      1.27
     365.6      98.447      0.55                      465.3      92.280      1.14
     370.7      98.269      0.61                      470.7      91.914      0.96
     375.0      98.119      0.62                      475.4      91.659      0.75
     380.0      97.927      0.64                      480.0      91.464      0.55
     385.1      97.732      0.67                      485.4      91.296      0.37
     390.0      97.529      0.69                      490.0      91.209      0.24
     395.6      97.278      0.75                      495.4      91.150      0.15
     400.5      97.041      0.82                      500.0      91.101      0.11
     405.4      96.786      0.88                      505.4      91.080      0.08
     410.3      96.514      0.92                      510.1      91.059      0.04
     415.2      96.231      0.98                      515.4      91.048      0.04
     420.1      95.884      1.06                      520.0      91.031      0.05
     425.8      95.533      1.16                      525.3      91.020      0.02
     430.5      95.173      1.22                      530.6      91.013      0.01
     435.3      94.797      1.28                      535.1      91.017      0.02
     440.0      94.406      1.34                      540.4      91.003      0.01
     445.6      93.924      1.37                      545.7      91.006      0.01
                                                      550.1      91.000      0.02
```

Sample: KAOLIN 45%
Size: 27.14 mg
Rate: 20C/MIN
Program: TGA Analysis V2.0

TGA

Date: 18-Sep-85 Time: 12:23:56
File: PEL-RESID.87 T.LYNCH.07
Operator: JT
Plotted: 18-Sep-85 13:07:19



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```
Sample: KAOLIN 45%                                  Date: 18-Sep-85    Time: 12:23:56
Size: 27.14 mg                                      File: PEL-RESID.84 T.LYNCH.07
Rate: 20C/MIN                                       Operator: JT
Program: General Analysis V1.0                      Plotted: 26-Sep-85    14:13:38

    Temp       Sig-A      Deriv-A                     Temp      Sig-A      Deriv-A
   [deg]        [%]       [%/min]                    [deg]       [%]       [%/min]

   150.7     100.048        0.01                     250.2     99.993        0.04
   155.5     100.048        0.02                     256.0     99.982        0.05
   160.3     100.041        0.01                     260.6     99.971        0.05
   165.1     100.044        0.01                     265.2     99.963        0.05
   171.0     100.041        0.03                     271.0     99.945        0.07
   175.8     100.033        0.02                     275.6     99.934        0.06
   180.5     100.029        0.00                     280.2     99.919        0.08
   185.3     100.029        0.01                     286.0     99.893        0.12
   190.1     100.026        0.02                     290.5     99.867        0.13
   196.0     100.026        0.02                     295.1     99.842        0.13
   200.7     100.018        0.02                     300.9     99.808        0.17
   205.5     100.018        0.00                     305.5     99.772        0.19
   210.3     100.018        0.01                     310.1     99.727        0.22
   215.1     100.015        0.02                     315.8     99.672        0.26
   221.0     100.011        0.02                     320.4     99.617        0.28
   225.7     100.007        0.00                     326.0     99.539        0.34
   230.4     100.011        0.01                     330.6     99.469        0.37
   235.1     100.004        0.02                     335.1     99.392        0.43
   240.9     100.000        0.01                     340.8     99.274        0.46
   245.8     100.000        0.01                     345.3     99.178        0.50
                                                     351.0     99.049        0.54
```

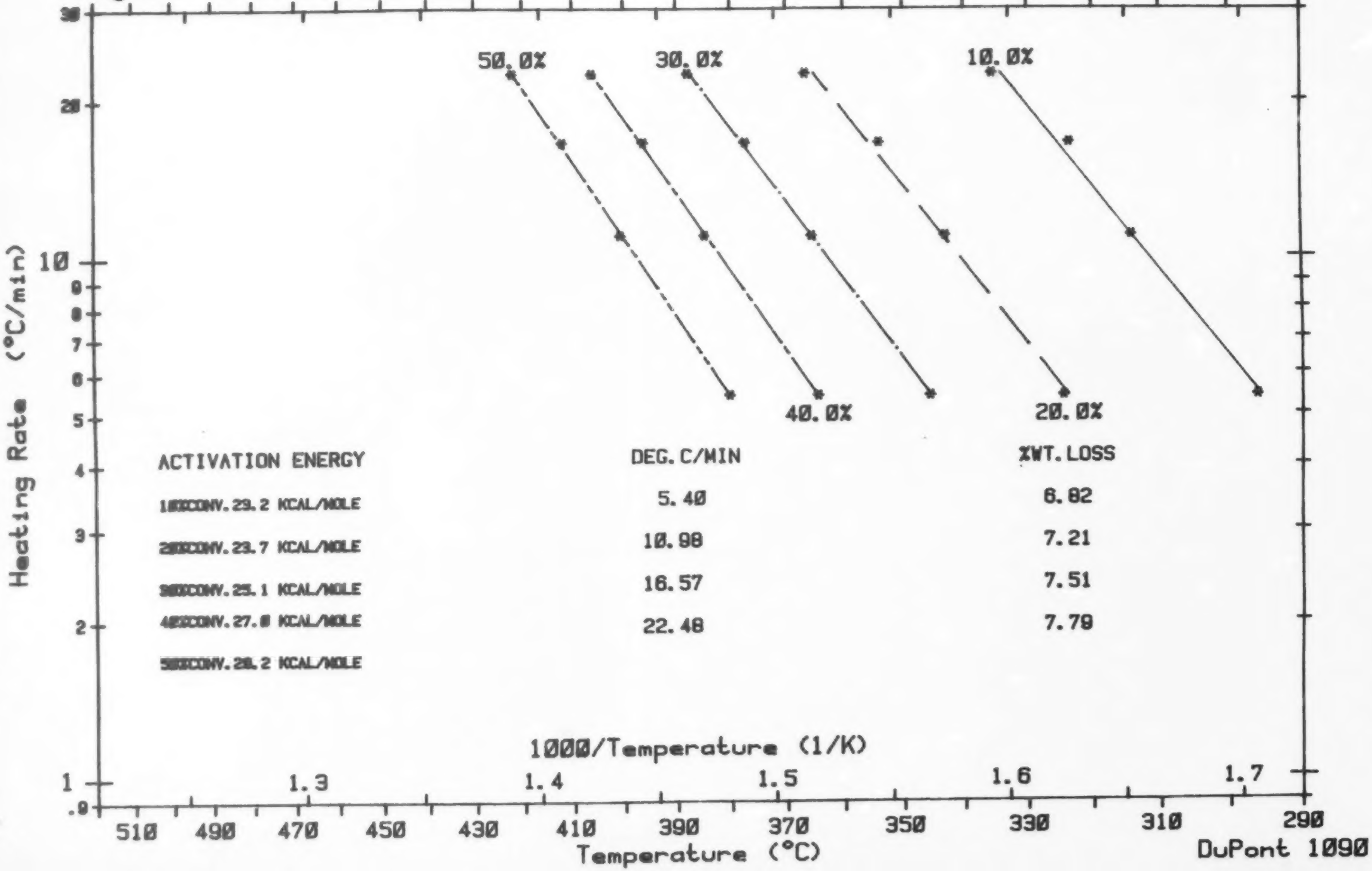
```
Sample: KAOLIN 45%                                    Date: 18-Sep-85   Time: 12:23:56
Size: 27.14 mg                                        File: PEL-RESID.84 T.LYNCH.07
Rate: 20C/MIN                                         Operator: JT
Program: General Analysis V1.0                        Plotted: 26-Sep-85   14:21:53

     Temp      Sig-A     Deriv-A                           Temp      Sig-A     Deriv-A
     [deg]      [%]      [%/min]                           [deg]      [%]      [%/min]

     351.0     99.049      0.54                            450.1     94.256      1.81
     355.5     98.935      0.59                            455.3     93.792      1.85
     360.0     98.817      0.61                            460.7     93.339      1.80
     365.6     98.655      0.66                            465.0     92.982      1.73
     370.0     98.523      0.70                            470.3     92.585      1.57
     375.6     98.342      0.75                            475.7     92.197      1.34
     380.0     98.187      0.80                            481.0     91.891      1.06
     385.6     97.985      0.81                            485.2     91.707      0.80
     390.0     97.819      0.86                            490.6     91.545      0.56
     395.6     97.598      0.91                            495.9     91.427      0.38
     401.1     97.362      0.97                            500.1     91.364      0.26
     405.5     97.163      1.04                            505.3     91.313      0.15
     411.0     96.891      1.12                            510.4     91.291      0.09
     415.4     96.658      1.19                            515.6     91.268      0.08
     420.[illegible] 96.[illegible] 1.[illegible]          520.7     91.250      0.05
     425.2     96.091      1.34                            525.9     91.235      0.03
     430.6     95.741      1.46                            530.0     91.232      0.01
     436.0     95.358      1.57                            535.1     91.232      0.02
     440.4     95.037      1.66                            540.2     91.221      0.02
     445.8     94.610      1.75                            545.3     91.224      0.01
                                                           550.4     91.221      0.00
```

Sample: 90% KAOLIN
FILE PEL-RESID.03

Program: TGA Kinetice V1.0

Date: 10-Jul-85 Time: 8:09:34

Operator: JT
Plotted: 10-Jul-85 10:22:32



ACTIVATION ENERGY
10%CONV.23.2 KCAL/MOLE
20%CONV.23.7 KCAL/MOLE
30%CONV.25.1 KCAL/MOLE
40%CONV.27.0 KCAL/MOLE
50%CONV.28.2 KCAL/MOLE

DEG.C/MIN	%WT.LOSS
5.40	6.82
10.98	7.21
16.57	7.51
22.48	7.79

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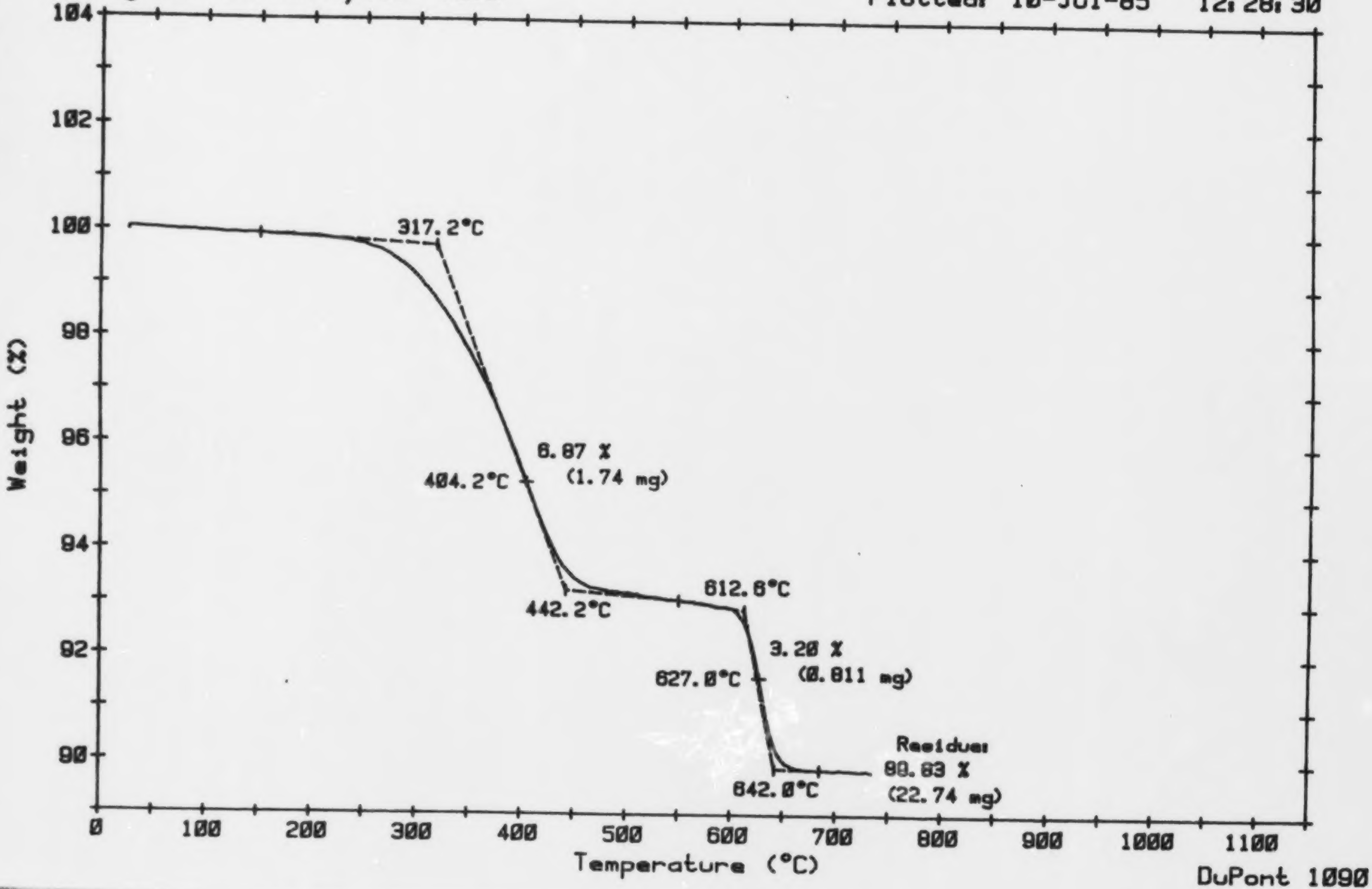
Sample: 90% KAOLIN
Size: 25.31 mg
Rate: 5C/MIN
Program: TGA Analysis V2.0

TGA

Date: 9-Jul-85 Time: 16:08:21
File: PEL-RESID.32 T.LYNCH.03
Operator: JT
Plotted: 10-Jul-85 12:28:30



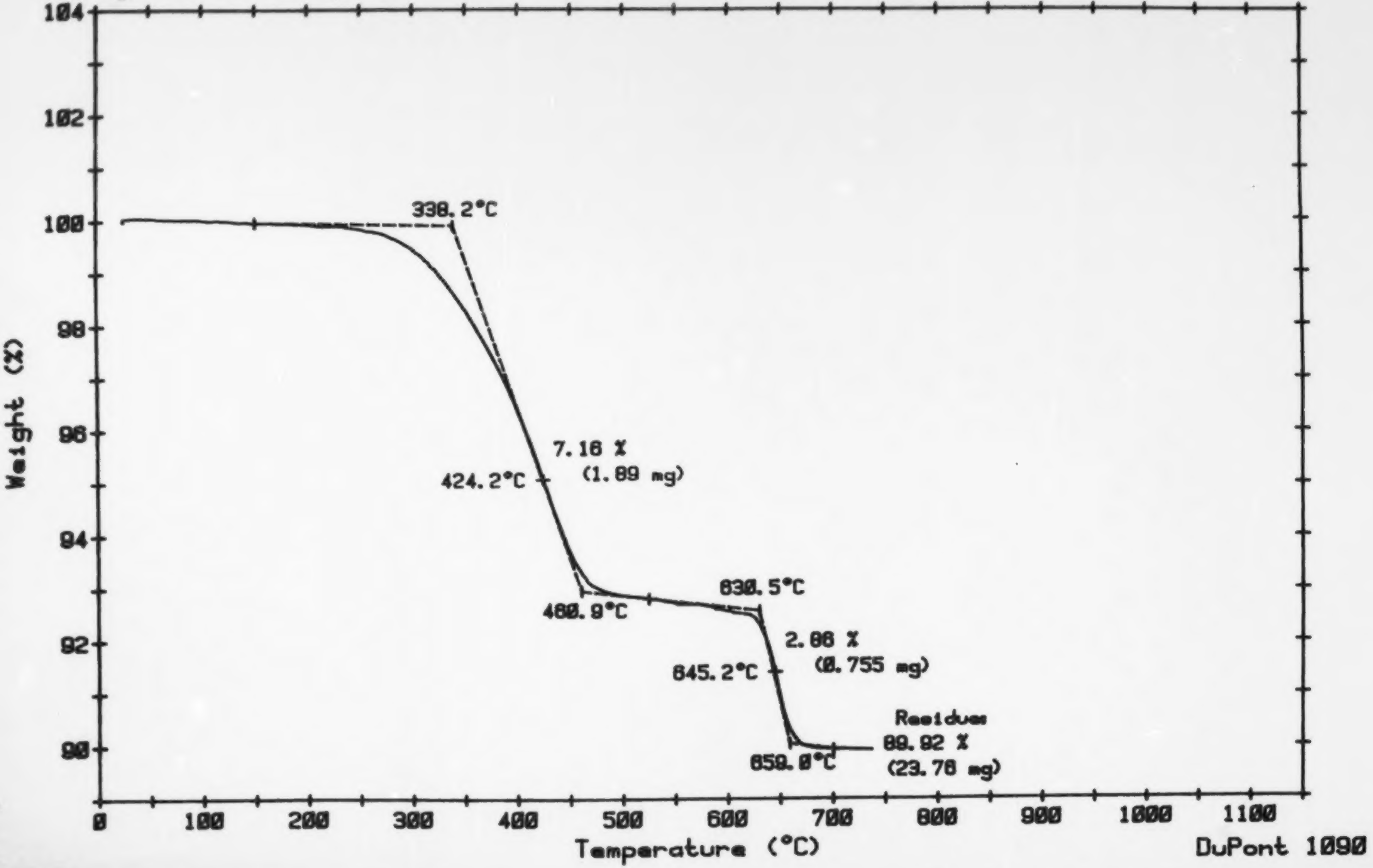
```
Sample: 90% KAOLIN                                    Date:   9-Jul-85    Time: 16:08:21
Size: 25.31 mg                                        File: PEL-RESID.32 T.LYNCH.03
Rate:  5C/MIN                                         Operator: JT
Program: General Analysis V1.0                        Plotted: 10-Jul-85    13:29:06

      Temp      Sig-A      Deriv-A                       Temp      Sig-A      Deriv-A
      [deg]      [%]       [%/min]                       [deg]      [%]       [%/min]

      150.1     99.933      0.01                         250.1     99.743      0.05
      155.3     99.920      0.01                         255.2     99.712      0.01
      160.2     99.913      0.01                         260.1     99.684      0.05
      165.1     99.917      0.04                         265.0     99.660      0.07
      170.0     99.921      0.02                         270.2     99.601      0.07
      175.1     99.933      0.03                         275.2     99.530      0.07
      180.1     99.913     -0.07                         280.0     99.471      0.09
      185.0     99.897      0.04                         285.1     99.411      0.09
      190.2     99.905      0.03                         290.2     99.328      0.10
      195.2     99.901     -0.01                         295.3     99.238      0.14
      200.1     99.888     -0.02                         300.2     99.135      0.12
      205.1     99.889     -0.02                         305.2     99.001      0.18
      210.0     99.885      0.04                         310.0     98.910      0.14
      215.1     99.858      0.01                         315.1     98.783      0.16
      220.1     99.854      0.02                         320.2     98.633      0.15
      225.0     99.842      0.01                         325.2     98.511      0.16
      230.2     99.834     -0.01                         330.2     98.353      0.18
      235.1     99.828      0.02                         335.0     98.203      0.22
      240.2     99.795      0.01                         340.2     98.017      0.19
      245.3     99.783      0.00                         345.2     97.847      0.18
                                                         350.2     97.683      0.22
```

```
Sample: 90% KAOLIN                                     Date:  9-Jul-85    Time: 16:08:21
Size: 25.31 mg                                         File: PEL-RESID.32 T.LYNCH.03
Rate:  5C/MIN                                          Operator: JT
Program: General Analysis V1.0                         Plotted: 10-Jul-85   13:37:17

      Temp       Sig-A      Deriv-A                       Temp       Sig-A      Deriv-A
      [deg]       [%]       [%/min]                       [deg]       [%]       [%/min]

      350.2     97.693        0.22                        450.0     93.478        0.09
      355.0     97.503        0.21                        455.2     93.407        0.05
      360.2     97.314        0.21                        460.1     93.355        0.05
      365.2     97.148        0.22                        465.2     93.304        0.06
      370.2     96.919        0.22                        470.2     93.280        0.02
      375.1     96.701        0.28                        475.0     93.269        0.03
      380.0     96.480        0.27                        480.2     93.245        0.03
      385.1     96.230        0.23                        485.0     93.241        0.07
      390.1     95.998        0.27                        490.0     93.225        0.01
      395.0     95.745        0.28                        495.2     93.205        0.01
      400.2     95.485        0.28                        500.1     93.209        0.00
      405.0     95.228        0.32                        505.2     93.193        0.02
      410.0     94.967        0.28                        510.1     93.178        0.03
      415.1     94.691        0.25                        515.0     93.150        0.00
      420.1     94.458        0.26                        520.2     93.130        0.00
      425.1     94.225        0.25                        525.0     93.130        0.00
      430.1     94.031        0.21                        530.0     93.114       -0.01
      435.1     93.833        0.18                        535.0     93.079        0.04
      440.0     93.683        0.14                        540.0     93.063        0.01
      445.1     93.565        0.10                        545.1     93.063        0.00
                                                          550.1     93.067        0.04
```

Sample: 90% KAOLIN.
Size: 26.43 mg
Rate: 10C/MIN
Program: TGA Analysis V2.0

TGA

Date: 9-Jul-85 Time: 8:43:05
File: PEL-RESID.33 T.LYNCH.03
Operator: JT
Plotted: 10-Jul-85 14:34:59



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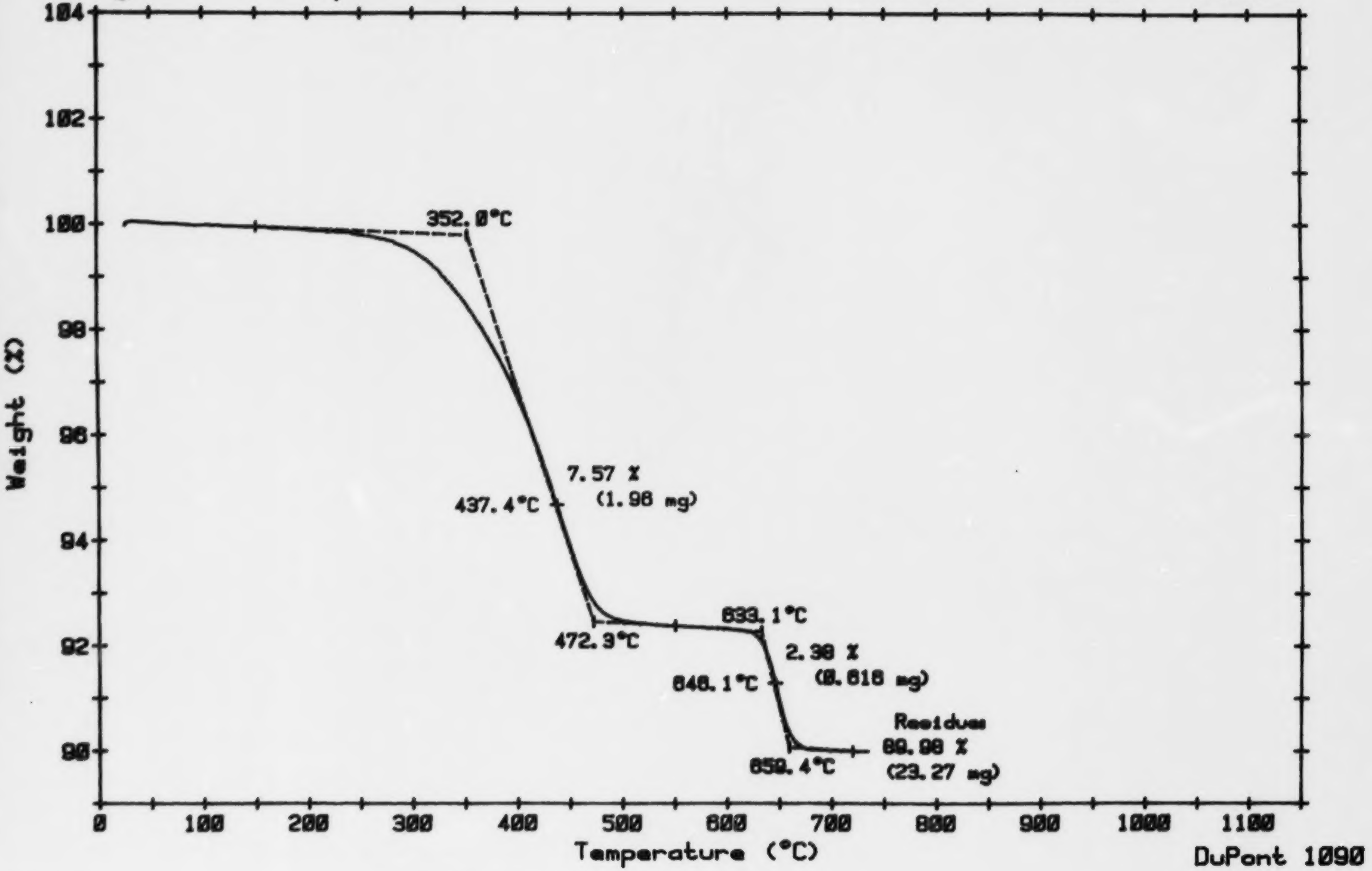
```
Sample: 90% KAOLIN.                                     Date:  9-Jul-85   Time:  8:43:05
Size: 26.43 mg                                          File: PEL-RESID.33 T.LYNCH.03
Rate: 10C/MIN                                           Operator: JT
Program: General Analysis V1.0                          Plotted: 10-Jul-85    15:48:08

      Temp       Sig-A      Deriv-A                           Temp       Sig-A      Deriv-A
      [deg]       [%]       [%/min]                           [deg]       [%]       [%/min]

      150.3     99.955        0.02                            250.4     99.830        0.06
      155.2     99.962       -0.03                            255.0     99.818        0.09
      160.0     99.962        0.01                            260.2     99.807        0.04
      165.4     99.955        0.04                            265.5     99.758        0.02
      170.1     99.943        0.02                            270.0     99.754        0.08
      175.3     99.951       -0.01                            275.2     99.720        0.13
      180.0     99.943        0.03                            280.4     99.663        0.09
      185.3     99.932        0.03                            285.0     99.625        0.10
      190.1     99.921       -0.01                            290.1     99.569        0.15
      195.4     99.924       -0.01                            295.2     99.504        0.13
      200.2     99.924        0.04                            300.4     99.432        0.20
      205.6     99.909        0.01                            305.5     99.353        0.15
      210.3     99.905        0.03                            310.1     99.273        0.23
      215.0     99.898        0.01                            315.3     99.175        0.27
      220.3     99.894        0.00                            320.3     99.060        0.22
      225.1     99.894       -0.01                            325.4     98.940        0.30
      230.4     99.894        0.04                            330.5     98.823        0.29
      235.2     99.875        0.00                            335.0     98.706        0.32
      240.4     99.868        0.07                            340.0     98.566        0.32
      245.1     99.845       -0.01                            345.0     98.426        0.32
                                                              350.0     98.282        0.31
```

```
Sample: 90% KAOLIN.                                   Date:   9-Jul-85    Time:   8:43:05
Size:  26.43 mg                                       File:  PEL-RESID.33 T.LYNCH.03
Rate:  10C/MIN                                        Operator:  JT
Program:  General Analysis V1.0                       Plotted:  10-Jul-85    15:56:55

      Temp       Sig-A      Deriv-A                        Temp       Sig-A      Deriv-A
      [deg]       [%]       [%/min]                        [deg]       [%]       [%/min]

      350.0     98.282        0.31                         450.0     93.677        0.47
      355.0     98.100        0.37                         455.3     93.461        0.36
      360.0     97.945        0.39                         460.2     93.313        0.34
      365.5     97.760        0.38                         465.4     93.186        0.29
      370.0     97.600        0.39                         470.0     93.064        0.17
      375.0     97.423        0.41                         475.3     93.007        0.12
      380.0     97.234        0.44                         480.0     92.969        0.08
      385.1     97.029        0.43                         485.2     92.927        0.05
      390.0     96.821        0.49                         490.5     92.897        0.06
      395.4     96.583        0.48                         495.1     92.874        0.04
      400.3     96.337        0.52                         500.3     92.859       -0.02
      405.1     96.102        0.55                         505.3     92.848        0.02
      410.0     95.850        0.56                         510.4     92.833        0.01
      415.1     95.584        0.61                         515.5     92.829        0.05
      420.5     95.277        0.63                         520.4     92.814        0.04
      425.3     94.994        0.58                         525.0     92.795        0.03
      430.2     94.721        0.64                         530.0     92.799       -0.01
      435.0     94.437        0.59                         535.4     92.791        0.10
      440.4     94.153        0.54                         540.3     92.734        0.03
      445.3     93.896        0.52                         545.3     92.731       -0.01
                                                           550.2     92.712        0.16
```

Sample: 90% KAOLIN.
Size: 25.87 mg
Rate: 15C/MIN
Program: TGA Analysis V2.0

TGA

Date: 9-Jul-85 Time: 11:16:06
File: PEL-RESID.34 T.LYNCH.03
Operator: JT
Plotted: 11-Jul-85 8:02:57



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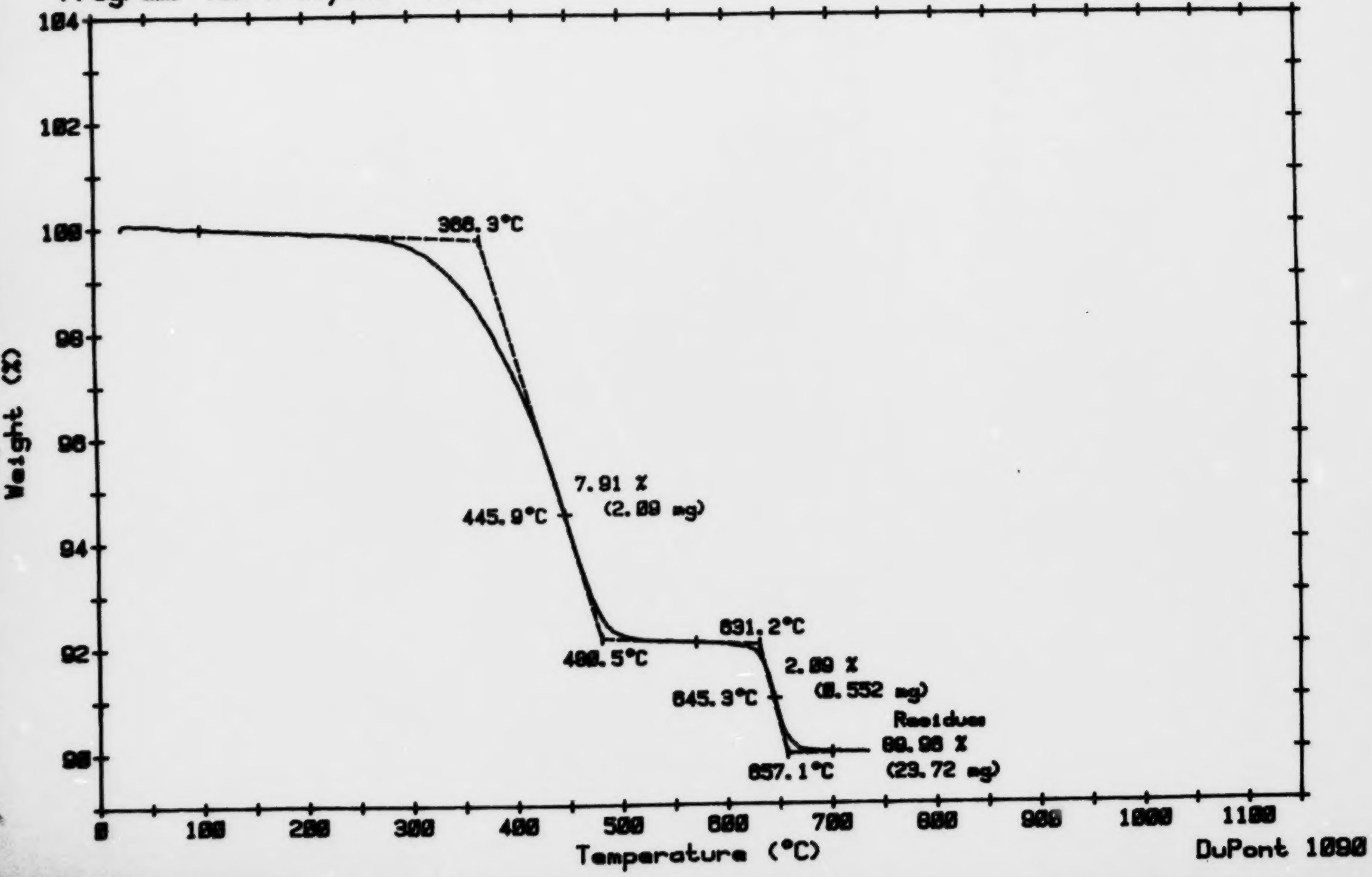
```
Sample: 90% KAOLIN.                                 Date:   9-Jul-85    Time: 11:16:06
Size: 25.87 mg                                      File: PEL-RESID.34 T.LYNCH.03
Rate: 15C/MIN                                       Operator: JT
Program: General Analysis V1.0                      Plotted: 11-Jul-85     8:36:14

     Temp       Sig-A      Deriv-A                   Temp       Sig-A      Deriv-A
     [deg]       [%]       [%/min]                   [deg]       [%]       [%/min]

     150.9      99.954       0.01                    250.1      99.811       0.05
     155.5      99.942      -0.01                    255.4      99.783       0.06
     160.0      99.950       0.03                    260.7      99.784       0.05
     165.4      99.934       0.00                    265.1      99.758       0.09
     170.6      99.923       0.05                    270.4      99.718       0.08
     175.4      99.927       0.02                    275.7      99.708       0.11
     180.6      99.923       0.02                    280.1      99.679       0.11
     185.3      99.915      -0.02                    285.4      99.644       0.15
     190.6      99.915      -0.01                    290.5      99.594       0.14
     195.3      99.907       0.04                    295.6      99.548       0.18
     200.7      99.892       0.03                    300.2      99.505       0.19
     205.3      99.892      -0.01                    305.3      99.439       0.24
     210.7      99.880       0.04                    310.4      99.378       0.24
     215.2      99.872       0.04                    315.6      99.265       0.30
     220.6      99.857       0.00                    320.7      99.188       0.31
     225.1      99.865       0.01                    325.0      99.118       0.39
     230.4      99.853       0.03                    330.1      99.000       0.40
     235.6      99.859       0.02                    335.3      98.875       0.41
     240.3      99.849       0.06                    340.4      98.743       0.40
     245.6      99.822       0.06                    345.5      98.627       0.43
                                                     350.6      98.488       0.47
```

```
Sample: 90% KAOLIN.                                    Date:  9-Jul-85    Time:  11:16:06
Size:  25.87 mg                                        File:  PEL-RESID.34  T.LYNCH.03
Rate:  15C/MIN                                         Operator:  JT
Program:  General Analysis V1.0                        Plotted:  11-Jul-85     8:47:34

      Temp        Sig-A      Deriv-A                         Temp        Sig-A      Deriv-A
      [deg]        [%]       [%/min]                         [deg]        [%]       [%/min]

      350.6       98.488       0.47                          450.1       93.881       0.91
      355.7       98.345       0.50                          455.6       93.583       0.91
      360.8       98.183       0.53                          460.3       93.311       0.76
      365.0       98.055       0.53                          465.1       93.095       0.67
      370.1       97.893       0.58                          470.5       92.894       0.40
      375.2       97.715       0.60                          475.1       92.755       0.30
      380.2       97.529       0.62                          480.5       92.654       0.28
      385.2       97.340       0.64                          485.2       92.585       0.16
      390.1       97.151       0.66                          490.5       92.538       0.13
      395.1       96.942       0.73                          495.2       92.503       0.08
      400.0       96.718       0.77                          500.6       92.476       0.06
      405.8       96.451       0.77                          505.3       92.469       0.05
      410.7       96.215       0.82                          510.8       92.438       0.07
      415.6       95.958       0.88                          515.3       92.438       0.02
      420.5       95.685       0.89                          520.7       92.438       0.02
      425.4       95.407       0.96                          525.3       92.422       0.04
      430.2       95.125       0.98                          530.6       92.407       0.03
      435.0       94.815       1.02                          535.3       92.399       0.01
      440.6       94.458       1.02                          540.6       92.384       0.03
      445.4       94.188       0.98                          545.1       92.388      -0.01
                                                             550.4       92.384       0.03
```

Sample: 90% KAOLIN.
Size: 26.37 mg
Rate: 20C/MIN
Program: TGA Analysis V2.0

TGA

Date: 10-Jul-85 Time: 8:09:34
File: PEL-RESID.35 T.LYNCH.03
Operator: JT
Plotted: 11-Jul-85 10:42:40



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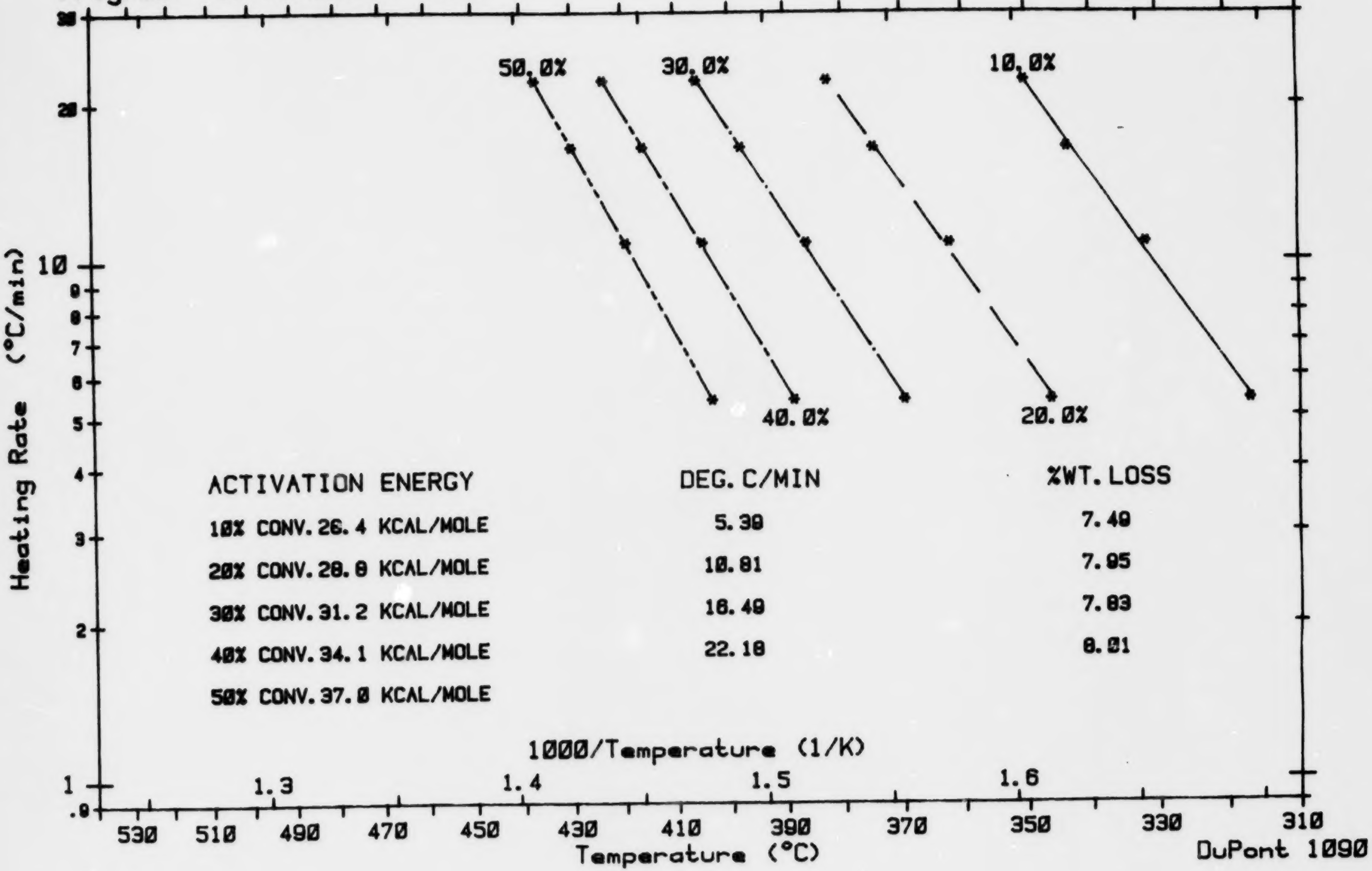
```
Sample: 90% KAOLIN.                                 Date: 10-Jul-85    Time:  8:09:34
Size: 26.37 mg                                      File: PEL-RESID.35 T.LYNCH.03
Rate: 20C/MIN                                       Operator: JT
Program: General Analysis V1.0                      Plotted: 11-Jul-85   11:12:13

     Temp       Sig-A      Deriv-A                       Temp       Sig-A      Deriv-A
     [deg]       [%]       [%/min]                       [deg]       [%]       [%/min]

     150.1     99.928        0.00                        250.0     99.829        0.07
     156.2     99.920        0.02                        255.9     99.795        0.06
     161.0     99.909        0.05                        260.7     99.791        0.03
     165.9     99.909        0.00                        265.4     99.772        0.08
     170.6     99.908       -0.02                        270.2     99.765        0.07
     175.5     99.913        0.03                        276.1     99.746        0.09
     180.2     99.898        0.01                        280.9     99.723        0.08
     185.0     99.905        0.00                        285.6     99.719        0.15
     191.1     99.896        0.02                        290.3     99.674        0.17
     195.9     99.894        0.07                        296.1     99.644        0.17
     200.8     99.879        0.05                        300.8     99.606        0.18
     205.6     99.867        0.03                        305.5     99.571        0.20
     210.5     99.867        0.01                        310.2     99.507        0.25
     215.3     99.863        0.00                        316.0     99.442        0.32
     220.1     99.867       -0.02                        320.7     99.367        0.39
     226.1     99.863        0.05                        325.3     99.295        0.38
     230.9     99.852        0.02                        330.0     99.211        0.42
     235.7     99.848        0.02                        335.8     99.094        0.46
     240.5     99.848        0.06                        340.4     98.995        0.48
     245.2     99.826        0.08                        345.0     98.904        0.50
                                                         350.8     98.775        0.62
```

```
Sample: 90% KAOLIN.                                   Date: 10-Jul-85    Time:   8:09:34
Size: 26.37 mg                                        File: PEL-RESID.35 T.LYNCH.03
Rate: 20C/MIN                                         Operator: JT
Program: General Analysis V1.0                        Plotted: 11-Jul-85    11:31:10

     Temp       Sig-A      Deriv-A                         Temp       Sig-A      Deriv-A
     [deg]       [%]       [%/min]                         [deg]       [%]       [%/min]

     350.8      98.775       0.82                          450.2      94.205       1.47
     355.5      98.635       0.58                          455.7      93.837       1.40
     360.1      98.525       0.65                          460.0      93.557       1.30
     365.9      98.350       0.77                          465.4      93.248       1.23
     370.4      98.191       0.75                          470.7      92.954       1.07
     375.0      98.047       0.76                          475.0      92.760       0.91
     380.7      97.831       0.94                          480.3      92.559       0.72
     385.2      97.637       0.88                          485.6      92.385       0.58
     390.8      97.417       0.91                          490.9      92.288       0.32
     395.3      97.243       0.92                          495.1      92.252       0.18
     400.8      97.012       0.98                          500.4      92.206       0.17
     405.3      96.807       1.03                          505.6      92.165       0.11
     410.8      96.553       1.06                          510.8      92.153       0.06
     415.2      96.336       1.10                          515.9      92.127       0.10
     420.7      96.041       1.14                          520.0      92.108       0.05
     425.2      95.824       1.21                          525.1      92.104       0.03
     430.7      95.498       1.32                          530.1      92.096       0.03
     435.1      95.221       1.38                          535.1      92.085       0.02
     440.5      94.868       1.47                          540.1      92.085      -0.04
     445.9      94.486       1.58                          545.1      92.100       0.04
                                                           550.2      92.085       0.04
```

Sample: 10%MONTMORILLINITE Date: 7-Oct-85 Time: 13:47:44

Operator: JT

Program: TGA Kinetics V1.0 Plotted: 8-Oct-85 15:01:44

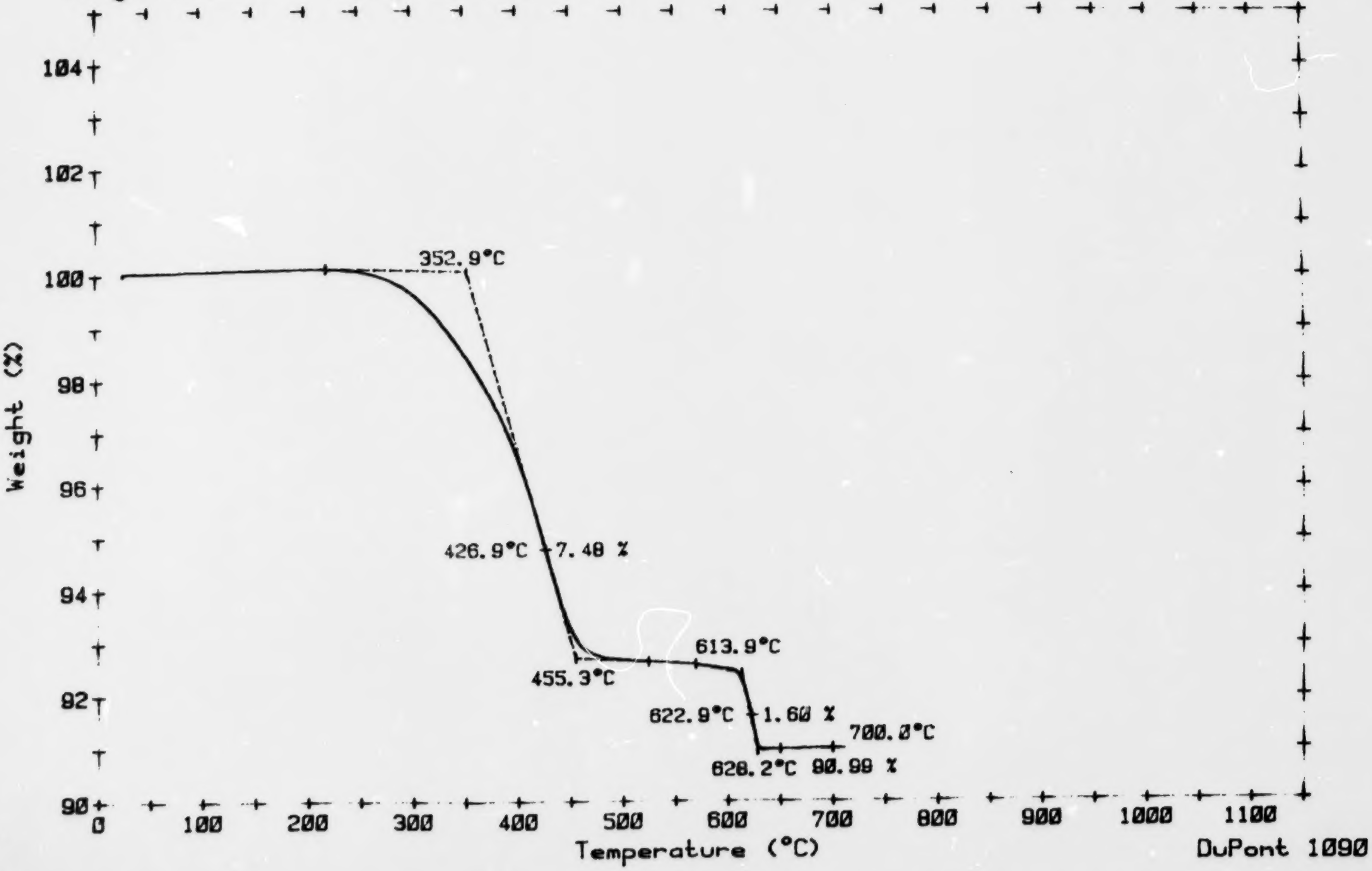


ACTIVATION ENERGY	DEG. C/MIN	%WT. LOSS
10% CONV. 26.4 KCAL/MOLE	5.38	7.48
20% CONV. 28.8 KCAL/MOLE	10.81	7.85
30% CONV. 31.2 KCAL/MOLE	16.48	7.83
40% CONV. 34.1 KCAL/MOLE	22.18	8.01
50% CONV. 37.0 KCAL/MOLE		

Sample: 10%MONTMORILLINITE
Size: 28.64 mg
Rate: 05C/MIN
Program: General Analysis V1.0

TGA

Date: 7-Oct-85 Time: 13:47:44
File: PEL/RESID.53 T.LYNCH.08
Operator: JT
Plotted: 7-Oct-85 16:01:37



DuPont 1090

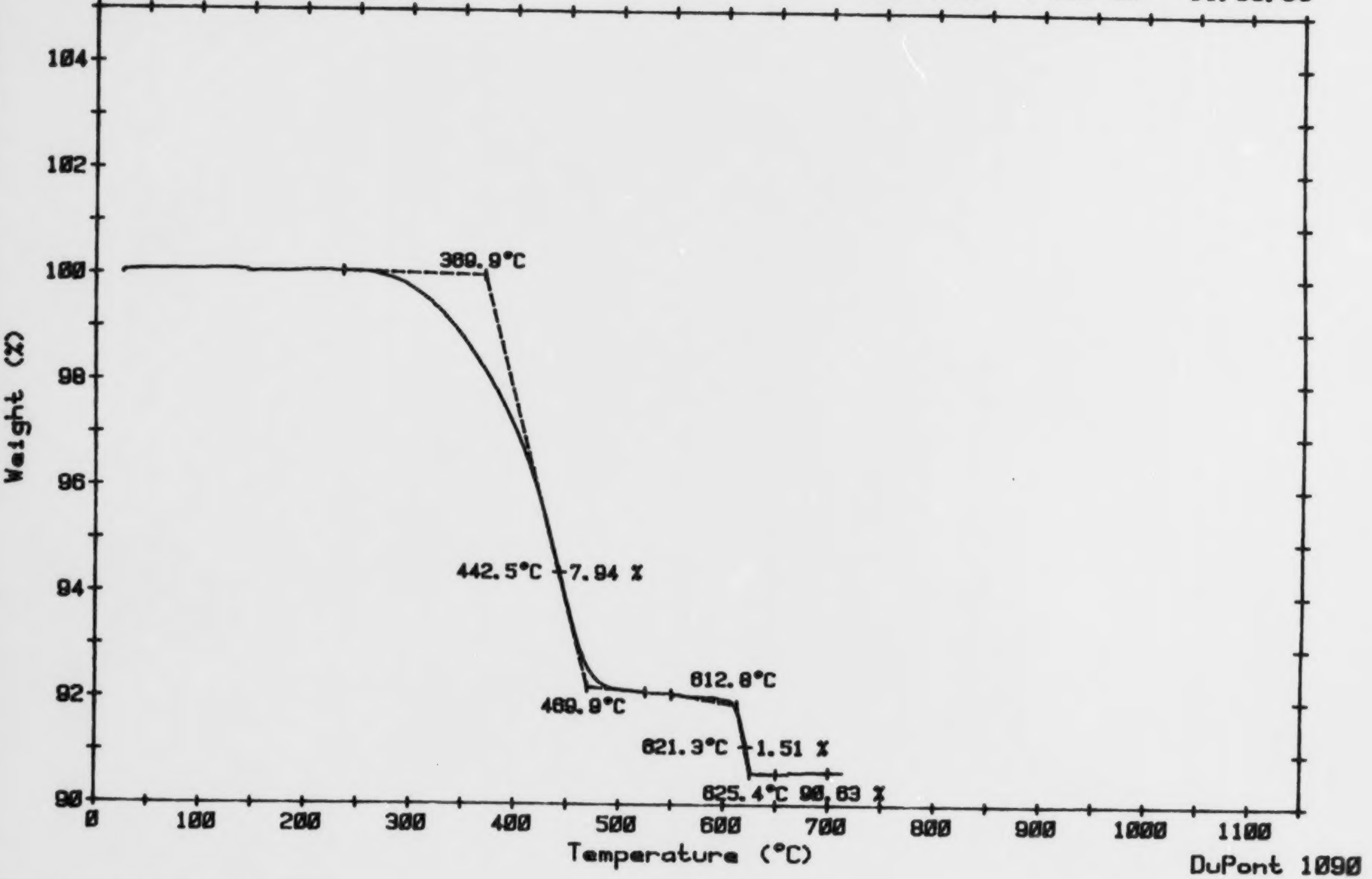
```
Sample: 10%MONTMORILLINITE                               Date:   7-Oct-85    Time: 13:47:44
Size: 28.64 mg                                           File: PEL/RESID.53 T.LYNCH.08
Rate: 05C/MIN                                            Operator: JT
Program: General Analysis V1.0                           Plotted:  9-Oct-85     9:06:42

      Temp       Sig-A      Deriv-A                            Temp       Sig-A      Deriv-A
      [deg]       [%]       [%/min]                            [deg]       [%]       [%/min]

      150.2     100.091      -0.01                             250.2     100.073      0.02
      155.1     100.091       0.01                             255.1     100.049      0.00
      160.2     100.094      -0.01                             260.1     100.028      0.03
      165.1     100.098      -0.01                             265.1     100.000      0.03
      170.1     100.101       0.00                             270.1      99.969      0.03
      175.1     100.098      -0.02                             275.0      99.934      0.04
      180.1     100.108       0.00                             280.0      99.895      0.06
      185.0     100.108       0.01                             285.1      99.846      0.05
      190.0     100.112      -0.02                             290.2      99.787      0.06
      195.1     100.115       0.01                             295.0      99.731      0.08
      200.1     100.108       0.00                             300.2      99.654      0.07
      205.1     100.119      -0.01                             305.1      99.570      0.09
      210.2     100.112       0.00                             310.1      99.476      0.10
      215.0     100.115      -0.02                             315.1      99.382      0.12
      220.1     100.115       0.02                             320.1      99.267      0.08
      225.0     100.108       0.00                             325.3      99.162      0.13
      230.2     100.108       0.01                             330.2      99.026      0.13
      235.1     100.101       0.00                             335.2      98.896      0.14
      240.2     100.084       0.02                             340.2      98.767      0.15
      245.2     100.077       0.02                             345.0      98.628      0.15
                                                               350.1      98.470      0.14
```

```
Sample: 10%MONTMORILLINITE                                  Date:  7-Oct-85   Time: 13:47:44
Size: 28.64 mg                                              File: PEL/RESID.53 T.LYNCH.08
Rate: 05C/MIN                                               Operator: JT
Program: General Analysis V1.0                              Plotted:  9-Oct-85    9:42:37

      Temp        Sig-A       Deriv-A                    Temp        Sig-A       Deriv-A
     [deg]         [%]        [%/min]                   [deg]         [%]        [%/min]

     350.1       98.470        0.14                     450.2       93.281        0.22
     355.0       98.334        0.19                     455.0       93.096        0.17
     360.1       98.160        0.18                     460.2       92.946        0.12
     365.0       98.002        0.16                     465.2       92.855        0.09
     370.1       97.824        0.19                     470.1       92.796        0.05
     375.0       97.657        0.21                     475.1       92.757        0.02
     380.1       97.454        0.21                     480.0       92.722        0.03
     385.0       97.248        0.23                     485.2       92.701        0.02
     390.1       97.021        0.24                     490.1       92.691        0.[illegible]
     395.2       96.780        0.27                     495.1       92.684        0.01
     400.0       96.518        0.28                     500.0       92.663        0.00
     405.1       96.238        0.31                     505.0       92.659       -0.02
     410.1       95.925        0.33                     510.2       92.649        0.01
     415.0       95.607        0.36                     515.0       92.642        0.01
     420.2       95.237        0.36                     520.0       92.638        0.01
     425.0       94.898        0.38                     525.0       92.631        0.01
     430.1       94.521        0.41                     530.1       92.628        0.02
     435.1       94.184        0.38                     535.1       92.621        0.00
     440.1       93.828        0.34                     540.1       92.621        0.00
     445.1       93.525        0.31                     545.1       92.610        0.02
                                                        550.0       92.600        0.01
```

Sample: 10%MONTMORILLINITE
Size: 24.99 mg
Rate: 10C/MIN
Program: General Analysis V1.0

TGA

Date: 4-Oct-85 Time: 10:31:29
File: PEL/RESID.50 T.LYNCH.08
Operator: JT
Plotted: 4-Oct-85 11:39:58



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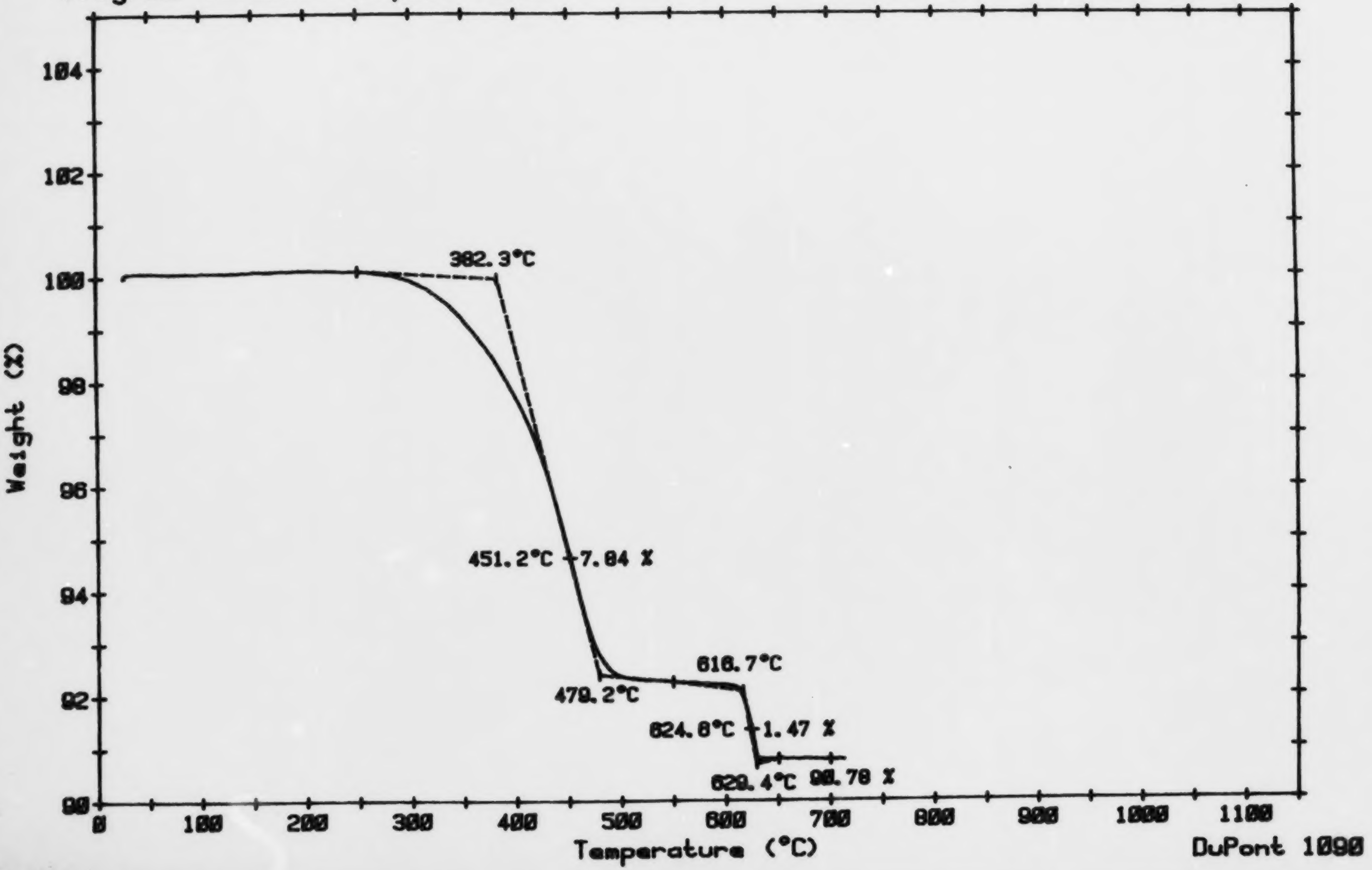
```
Sample: 10%MONTMORILLINITE                        Date:   4-Oct-85    Time: 10:31:29
Size: 24.99 mg                                    File: PEL/RESID.50 T.LYNCH.08
Rate: 10C/MIN                                     Operator: JT
Program: General Analysis V1.0                    Plotted:  8-Oct-85    16:05:55

     Temp       Sig-A      Deriv-A                     Temp       Sig-A      Deriv-A
     [deg]       [%]       [%/min]                     [deg]       [%]       [%/min]

     150.0     100.048      -0.03                      250.5     100.064      -0.02
     155.0     100.052      -0.01                      255.0     100.072       0.01
     160.3     100.060      -0.02                      260.4     100.060       0.02
     165.4     100.064       0.01                      265.5     100.032       0.07
     170.5     100.064      -0.01                      270.4     100.008       0.05
     175.0     100.064      -0.01                      275.3      99.988       0.07
     180.1     100.060       0.01                      280.4      99.956       0.08
     185.1     100.060       0.00                      285.3      99.924       0.07
     190.2     100.064      -0.02                      290.3      99.884       0.10
     195.3     100.072       0.00                      295.3      99.836       0.12
     200.2     100.076      -0.03                      300.2      99.780       0.14
     205.4     100.072       0.01                      305.2      99.708       0.15
     210.4     100.080      -0.02                      310.0      99.636       0.14
     215.4     100.088      -0.01                      315.5      99.560       0.17
     220.4     100.084       0.01                      320.5      99.476       0.21
     225.0     100.080       0.02                      325.4      99.388       0.21
     230.1     100.072       0.01                      330.3      99.288       0.26
     235.5     100.068       0.01                      335.1      99.172       0.24
     240.4     100.068       0.00                      340.1      99.064       0.25
     245.5     100.068       0.00                      345.0      98.940       0.27
                                                       350.4      98.808       0.29
```

```
Sample: 10%MONTMORILLINITE                                  Date:   4-Oct-85   Time: 10:31:29
Size: 24.99 mg                                              File: PEL/RESID.50 T.LYNCH.08
Rate: 10C/MIN                                               Operator: JT
Program: General Analysis V1.0                              Plotted:  8-Oct-85   16:15:25

     Temp       Sig-A      Deriv-A                    Temp       Sig-A      Deriv-A
     [deg]       [%]      [%/min]                     [deg]       [%]      [%/min]

     350.4     98.808       0.29                      450.0     93.803       0.79
     355.3     98.664       0.34                      455.2     93.418       0.75
     360.2     98.520       0.30                      460.4     93.062       0.66
     365.1     98.372       0.33                      465.1     92.802       0.51
     370.5     98.208       0.34                      470.3     92.582       0.37
     375.4     98.052       0.36                      475.1     92.442       0.25
     380.1     97.883       0.37                      480.4     92.334       0.18
     385.1     97.711       0.41                      485.1     92.270       0.11
     390.4     97.503       0.42                      490.2     92.222       0.05
     395.2     97.303       0.44                      495.2     92.202       0.03
     400.0     97.085       0.48                      500.5     92.178       0.05
     405.5     96.843       0.54                      505.1     92.166      -0.01
     410.2     96.595       0.55                      510.1     92.166       0.00
     415.1     96.335       0.62                      515.4     92.150       0.05
     420.4     96.007       0.67                      520.4     92.138       0.01
     425.3     95.703       0.73                      525.5     92.130       0.04
     430.5     95.327       0.78                      530.1     92.118       0.03
     435.3     94.959       0.83                      535.1     92.114       0.01
     440.4     94.551       0.79                      540.3     92.110       0.02
     445.2     94.183       0.89                      545.3     92.110       0.02
                                                      550.4     92.102       0.04
```

Sample: 10%MONTMORILLINITE
Size: 27.67 mg
Rate: 15C/MIN
Program: General Analysis V1.0

TGA

Date: 4-Oct-85 Time: 14:19:25
File: PEL/RESID.51 T.LYNCH.08
Operator: JT
Plotted: 4-Oct-85 15:29:05



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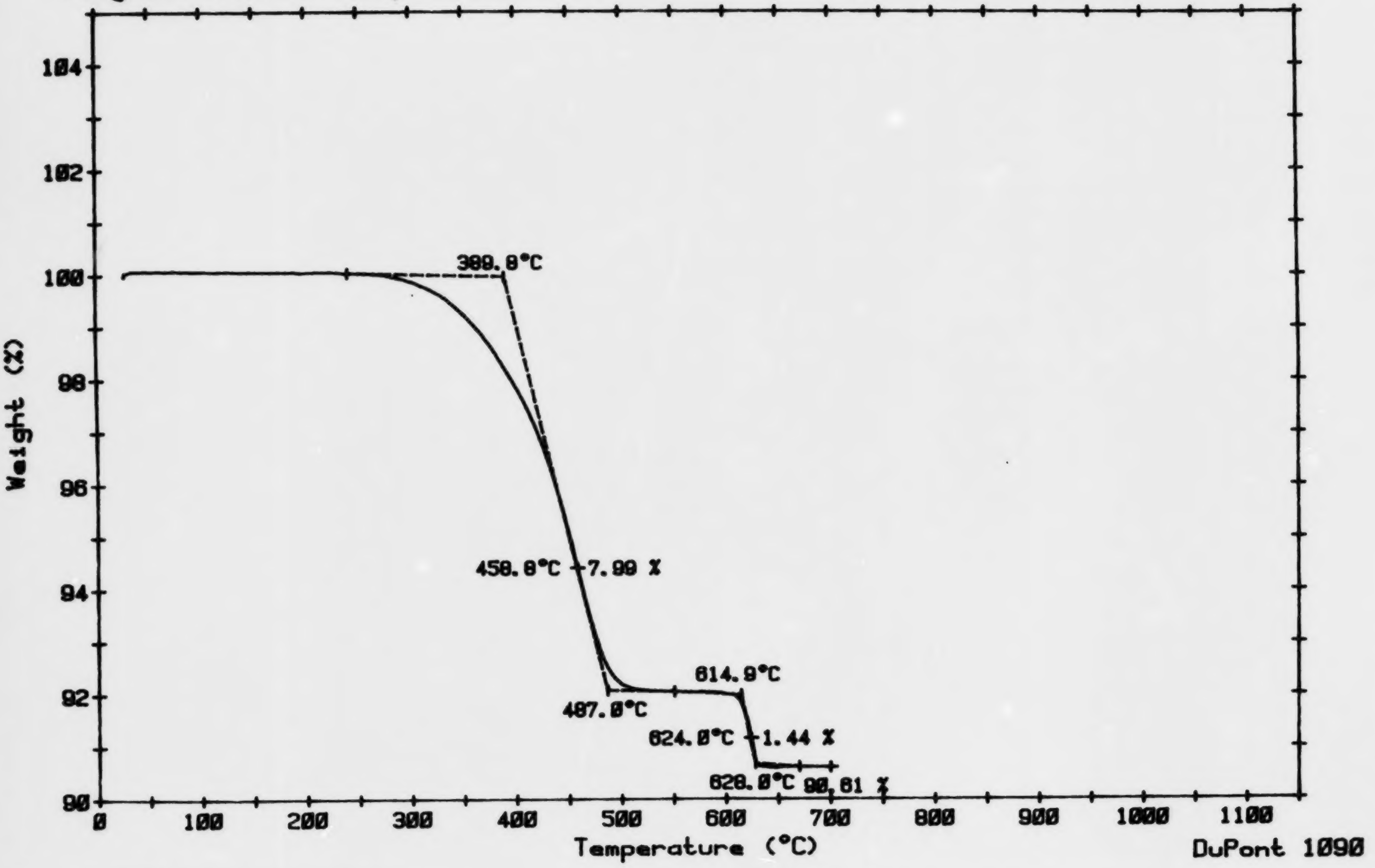
```
Sample: 10%MONTMORILLINITE                               Date:  4-Oct-85    Time: 14:19:25
Size: 27.67 mg                                           File: PEL/RESID.51 T.LYNCH.08
Rate: 15C/MIN                                            Operator: JT
Program: General Analysis V1.0                           Plotted:  9-Oct-85     8:14:01

     Temp       Sig-A       Deriv-A                       Temp       Sig-A       Deriv-A
     [deg]       [%]        [%/min]                       [deg]       [%]        [%/min]

     150.3     100.087       -0.03                        250.4     100.094        0.02
     155.8     100.094        0.02                        255.5     100.087        0.03
     160.2     100.090        0.01                        260.6     100.072        0.04
     165.6     100.094       -0.03                        265.8     100.058        0.07
     170.8     100.094       -0.02                        270.1     100.051        0.03
     175.3     100.105       -0.01                        275.3     100.033        0.07
     180.6     100.105        0.00                        280.3     100.025        0.03
     185.1     100.112       -0.01                        285.4     100.000        0.08
     190.3     100.105       -0.02                        290.5      99.967        0.11
     195.6     100.105        0.00                        295.5      99.946        0.10
     200.1     100.112       -0.03                        300.6      99.910        0.14
     205.3     100.116        0.03                        305.7      99.855        0.18
     210.5     100.105        0.00                        310.7      99.805        0.16
     215.7     100.101        0.00                        315.7      99.758        0.21
     220.0     100.108       -0.01                        320.8      99.682        0.23
     225.4     100.108        0.02                        325.8      99.606        0.26
     230.6     100.101        0.02                        330.0      99.545        0.23
     235.7     100.090       -0.03                        335.8      99.454        0.30
     240.0     100.101        0.02                        340.0      99.371        0.34
     245.2     100.094        0.01                        345.0      99.270        0.35
                                                          350.8      99.136        0.43
```

```
Sample: 10%MONTMORILLINITE                              Date:   4-Oct-85   Time: 14:19:25
Size: 27.67 mg                                          File: PEL/RESID.51 T.LYNCH.08
Rate: 15C/MIN                                           Operator: JT
Program: General Analysis V1.0                          Plotted:  9-Oct-85    8:22:53

      Temp       Sig-A     Deriv-A                     Temp       Sig-A     Deriv-A
      [deg]       [%]      [%/min]                     [deg]       [%]      [%/min]

      350.8      99.136      0.43                      450.4      94.676      1.25
      355.7      99.017      0.38                      455.2      94.304      1.24
      360.7      98.890      0.42                      460.7      93.867      1.23
      365.6      98.771      0.42                      465.4      93.520      1.08
      370.5      98.645      0.44                      470.1      93.216      0.94
      375.5      98.504      0.48                      475.6      92.920      0.72
      380.4      98.352      0.54                      480.3      92.732      0.55
      385.4      98.186      0.56                      485.0      92.591      0.40
      390.2      98.016      0.58                      490.5      92.472      0.28
      395.1      97.835      0.60                      495.1      92.410      0.16
      400.7      97.629      0.62                      500.6      92.363      0.10
      405.6      97.434      0.70                      505.3      92.334      0.06
      410.5      97.221      0.73                      510.7      92.320      0.03
      415.3      96.989      0.80                      515.3      92.313      0.04
      420.1      96.740      0.86                      520.7      92.298      0.05
      425.0      96.476      0.95                      525.2      92.284     -0.01
      430.6      96.126      1.04                      530.5      92.284      0.02
      435.3      95.808      1.10                      535.1      92.287      0.01
      440.1      95.469      1.14                      540.4      92.280      0.01
      445.7      95.049      1.21                      545.6      92.266      0.02
                                                       550.1      92.258      0.04
```

Sample: 10%MONTMORILLINITE
Size: 27.15 mg
Rate: 20C/MIN
Program: General Analysis V1.0

TGA

Date: 4-Oct-85 Time: 16:01:08
File: PEL/RESID.52 T.LYNCH.08
Operator: JT
Plotted: 4-Oct-85 16:41:00



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```
Sample: 10%MONTMORILLINITE                              Date:   4-Oct-85    Time: 16:01:08
Size: 27.15 mg                                          File: PEL/RESID.52 T.LYNCH.08
Rate: 20C/MIN                                           Operator: JT
Program: General Analysis V1.0                          Plotted:  9-Oct-85     8:34:14

      Temp       Sig-A       Deriv-A                       Temp       Sig-A       Deriv-A
      [deg]       [%]        [%/min]                       [deg]       [%]        [%/min]

      150.1     100.066        0.00                        250.9     100.044        0.04
      156.1     100.066        0.00                        255.5     100.033        0.04
      160.9     100.063        0.02                        260.1     100.026        0.03
      165.6     100.059        0.00                        265.8     100.029        0.02
      170.3     100.063        0.01                        270.4     100.015        0.05
      175.0     100.059       -0.01                        276.1     100.007        0.08
      180.9     100.059        0.01                        280.7      99.985        0.11
      185.6     100.059       -0.01                        285.3      99.963        0.11
      190.3     100.063        0.02                        291.0      99.937        0.12
      196.1     100.052        0.02                        295.5      99.912        0.13
      200.8     100.055       -0.03                        300.1      99.886        0.15
      205.5     100.063       -0.01                        305.8      99.845        0.17
      210.2     100.059        0.01                        310.3      99.805        0.19
      216.1     100.063        0.00                        316.0      99.757        0.22
      220.8     100.055       -0.01                        320.5      99.713        0.23
      225.4     100.066        0.01                        326.0      99.654        0.27
      230.0     100.055        0.04                        330.5      99.585        0.31
      235.9     100.048        0.03                        335.0      99.532        0.35
      240.5     100.044        0.00                        340.6      99.436        0.38
      245.1     100.041        0.00                        345.1      99.359        0.40
                                                           350.7      99.256        0.43
```

```
Sample: 10%MONTMORILLINITE                          Date:   4-Oct-85    Time: 16:01:08
Size: 27.15 mg                                      File: PEL/RESID.52 T.LYNCH.08
Rate: 20C/MIN                                       Operator: JT
Program: General Analysis V1.0                      Plotted:  9-Oct-85     8:49:58

     Temp       Sig-A      Deriv-A                     Temp       Sig-A      Deriv-A
     [deg]       [%]       [%/min]                     [deg]       [%]       [%/min]

     350.7     99.256        0.43                      450.2     95.119        1.87
     355.1     99.168        0.47                      455.6     94.696        1.76
     360.7     99.046        0.51                      460.9     94.250        1.76
     365.2     98.939        0.53                      465.1     93.900        1.71
     370.7     98.799        0.59                      470.4     93.484        1.57
     375.2     98.685        0.60                      475.7     93.119        1.37
     380.7     98.527        0.70                      480.9     92.802        1.11
     385.1     98.372        0.73                      485.1     92.604        0.90
     390.6     98.195        0.72                      490.3     92.416        0.63
     396.1     98.018        0.74                      495.5     92.283        0.44
     400.4     97.867        0.76                      500.8     92.195        0.25
     405.9     97.665        0.85                      506.0     92.150        0.18
     410.3     97.492        0.92                      510.1     92.117        0.11
     415.8     97.248        0.99                      515.3     92.095        0.06
     420.1     97.046        1.07                      520.4     92.080        0.04
     425.5     96.766        1.18                      525.6     92.080        0.03
     430.9     96.460        1.28                      530.8     92.066        0.05
     435.2     96.195        1.37                      535.8     92.062        0.02
     440.6     95.841        1.46                      540.8     92.062        0.00
     445.9     95.455        1.63                      545.9     92.058       -0.01
                                                       550.9     92.058        0.02
```

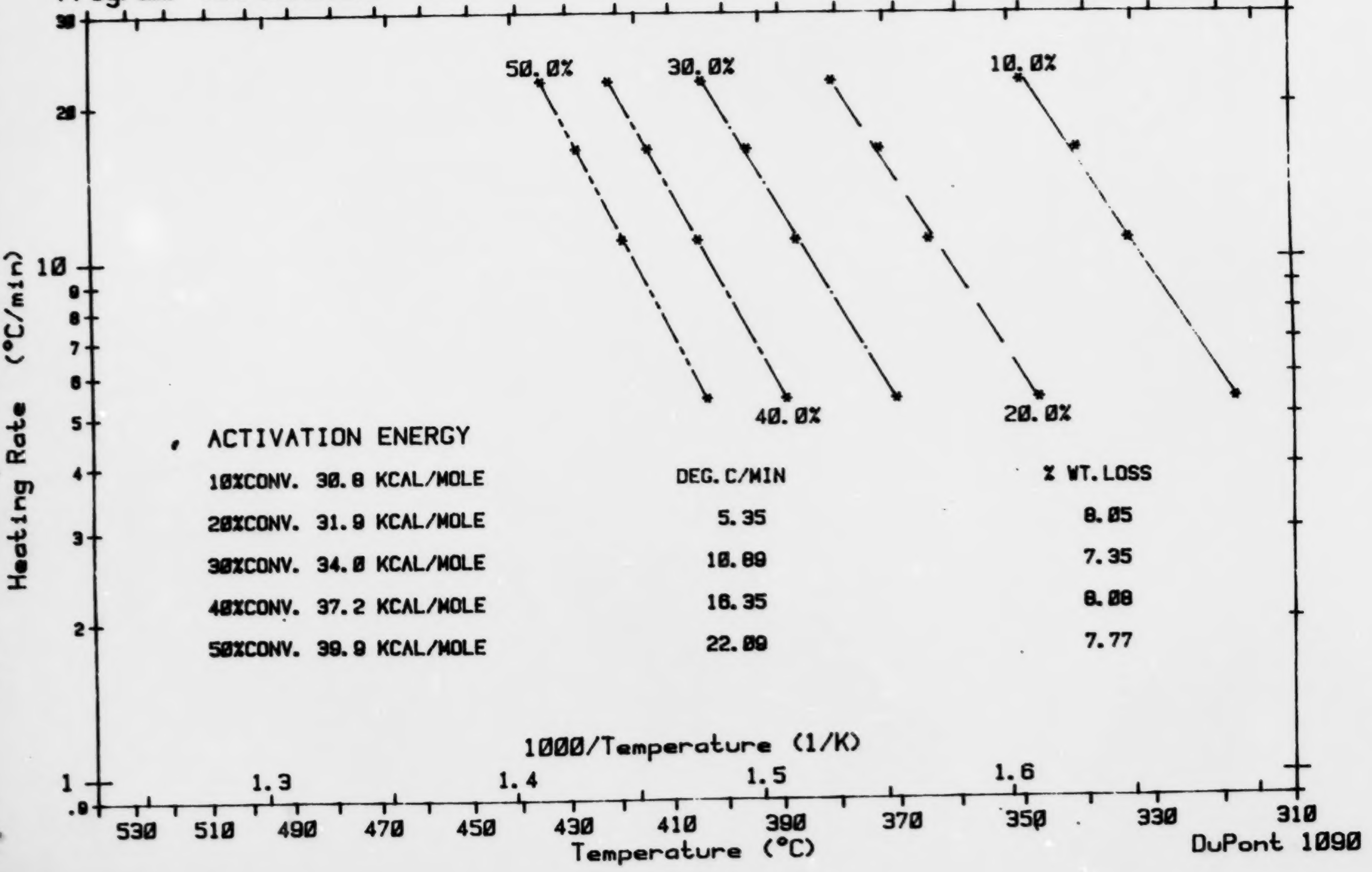
Sample: 10%MONT.80%SAND

Date: 22-Aug-85 Time: 16:12:36

Operator: JT

Program: TGA Kinetics V1.0

Plotted: 23-Aug-85 8:08:29



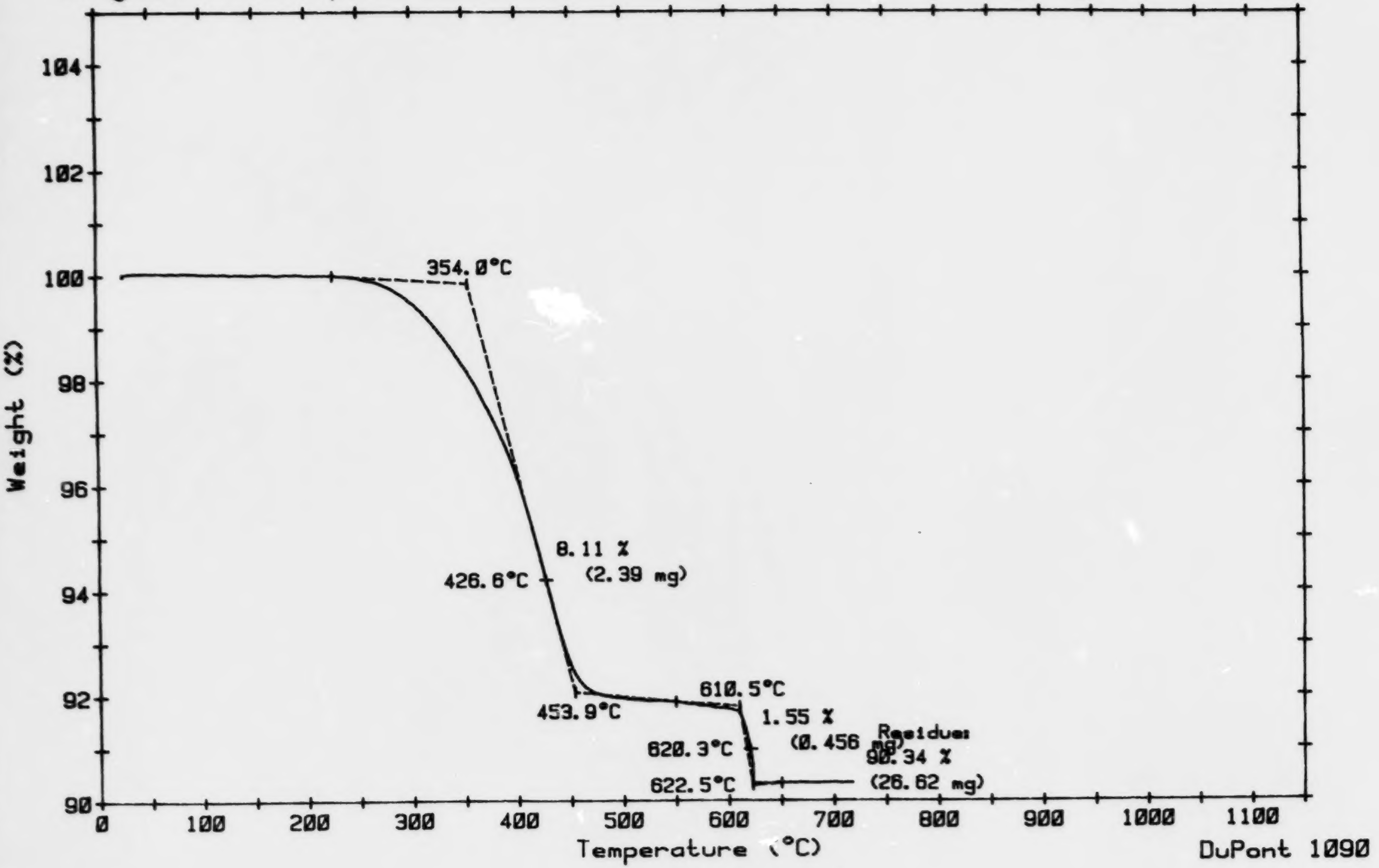
```
ACTIVATION ENERGY
10%CONV.  30.8 KCAL/MOLE          DEG.C/MIN          % WT.LOSS
20%CONV.  31.9 KCAL/MOLE             5.35               8.05
30%CONV.  34.0 KCAL/MOLE            10.89               7.35
40%CONV.  37.2 KCAL/MOLE            16.35               8.08
50%CONV.  39.9 KCAL/MOLE            22.09               7.77
```

DuPont 1090

Sample: 10%MONT. 80%SAND
Size: 29.47 mg
Rate: 05C/MIN
Program: TGA Analysis V2.0

TGA

Date: 21-Aug-85 Time: 15:54:43
File: PEL-RESID.70 T.LYNCH.05
Operator: JT
Plotted: 22-Aug-85 8:01:55



DuPont 1090

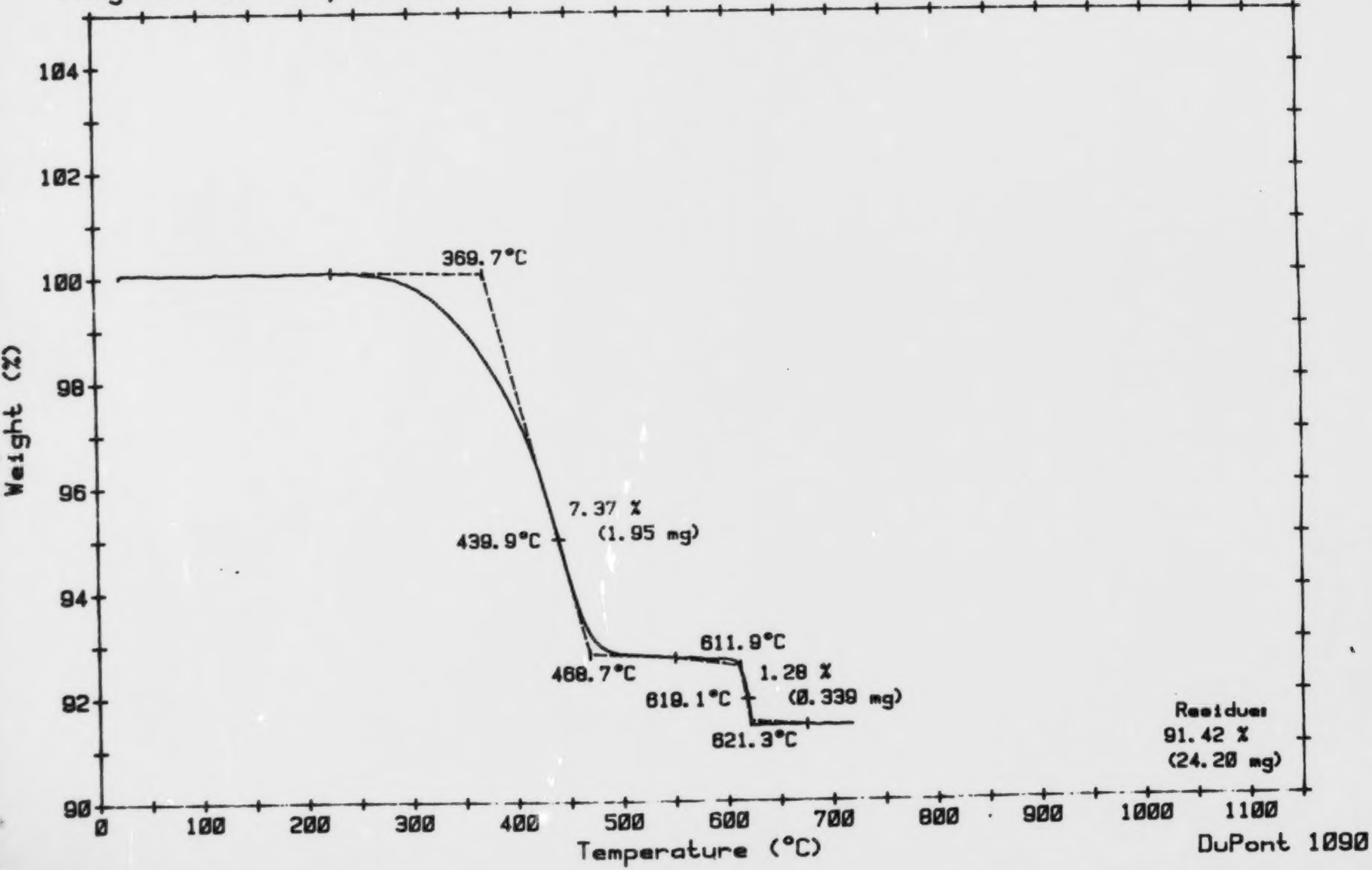
```
Sample: 10%MONT. 80%SAND                                  Date: 21-Aug-85    Time: 15:54:43
Size: 29.47 mg                                            File: PEL-RESID.70 T.LYNCH.05
Rate: 05C/MIN                                             Operator: JT
Program: General Analysis V1.0                            Plotted: 23-Aug-85    14:30:21

   Temp       Sig-A      Deriv-A                             Temp       Sig-A      Deriv-A
   [deg]      [%]        [%/min]                             [deg]      [%]        [%/min]

   200.1     100.003      -0.02                              300.0      99.491       0.09
   205.3     100.007      -0.01                              305.1      99.406       0.13
   210.2     100.010       0.00                              310.1      99.297       0.13
   215.0     100.010       0.00                              315.0      99.196       0.13
   220.0     100.003       0.03                              320.0      99.090       0.13
   225.1      99.997       0.00                              325.2      98.961       0.13
   230.0      99.997       0.02                              330.1      98.833       0.15
   235.0      99.983       0.01                              335.0      98.700       0.16
   240.2      99.973       0.03                              340.2      98.544       0.16
   245.1      99.956       0.01                              345.0      98.395       0.15
   250.2      99.939       0.03                              350.1      98.252       0.16
   255.1      99.915       0.01                              355.2      98.086       0.19
   260.1      99.905       0.01                              360.1      97.913       0.17
   265.2      99.874       0.03                              365.2      97.713       0.23
   270.1      99.837       0.07                              370.1      97.519       0.25
   275.2      99.800       0.06                              375.0      97.332       0.21
   280.2      99.752       0.03                              380.0      97.132       0.25
   285.2      99.695       0.06                              385.2      96.898       0.23
   290.3      99.637       0.08                              390.1      96.667       0.26
   295.0      99.579       0.08                              395.0      96.409       0.31
                                                             400.1      96.111       0.33
```

```
Sample: 10%MONT.80%SAND                         Date: 21-Aug-85    Time: 15:54:43
Size: 29.47 mg                                  File: PEL-RESID.70 T.LYNCH.05
Rate: 05C/MIN                                   Operator: JT
Program: General Analysis V1.0                  Plotted: 23-Aug-85    14:40:45

     Temp      Sig-A     Deriv-A                  Temp      Sig-A     Deriv-A
     [deg]      [%]     [%/min]                   [deg]      [%]     [%/min]

     400.1    96.111      0.33                    500.2    91.963      0.00
     405.1    95.788      0.35                    505.0    91.946     -0.02
     410.2    95.452      0.38                    510.0    91.929     -0.02
     415.3    95.079      0.39                    515.0    91.929     -0.01
     420.1    94.719      0.39                    520.2    91.919      0.00
     425.0    94.336      0.40                    525.1    91.906      0.00
     430.1    93.935      0.42                    530.1    91.902     -0.01
     435.2    93.538      0.43                    535.1    91.909      0.03
     440.3    93.172      0.35                    540.1    91.899     -0.01
     445.0    92.849      0.32                    545.0    91.892     -0.01
     450.2    92.581      0.25                    550.0    91.885      0.01
     455.0    92.388      0.16                    555.1    91.875      0.02
     460.1    92.242      0.12                    560.1    91.851      0.03
     465.1    92.133      0.07                    565.0    91.838      0.01
     470.0    92.086      0.04                    570.1    91.817      0.03
     475.0    92.045      0.04                    575.0    91.811      0.05
     480.1    92.007      0.03                    580.1    91.794      0.02
     485.1    91.987      0.01                    585.2    91.797      0.02
     490.1    91.974     -0.02                    590.0    91.766      0.02
     495.0    91.967      0.03                    595.0    91.760      0.00
                                                  600.2    91.750      0.04
```

Sample: 10%MONT. 80%SAND
Size: 26.47 mg
Rate: 10C/MIN
Program: TGA Analysis V2.0

TGA

Date: 22-Aug-85 Time: 8:36:05
File: PEL-RESID.71 T.LYNCH.05
Operator: JT
Plotted: 22-Aug-85 10:00:34



DuPont 1090

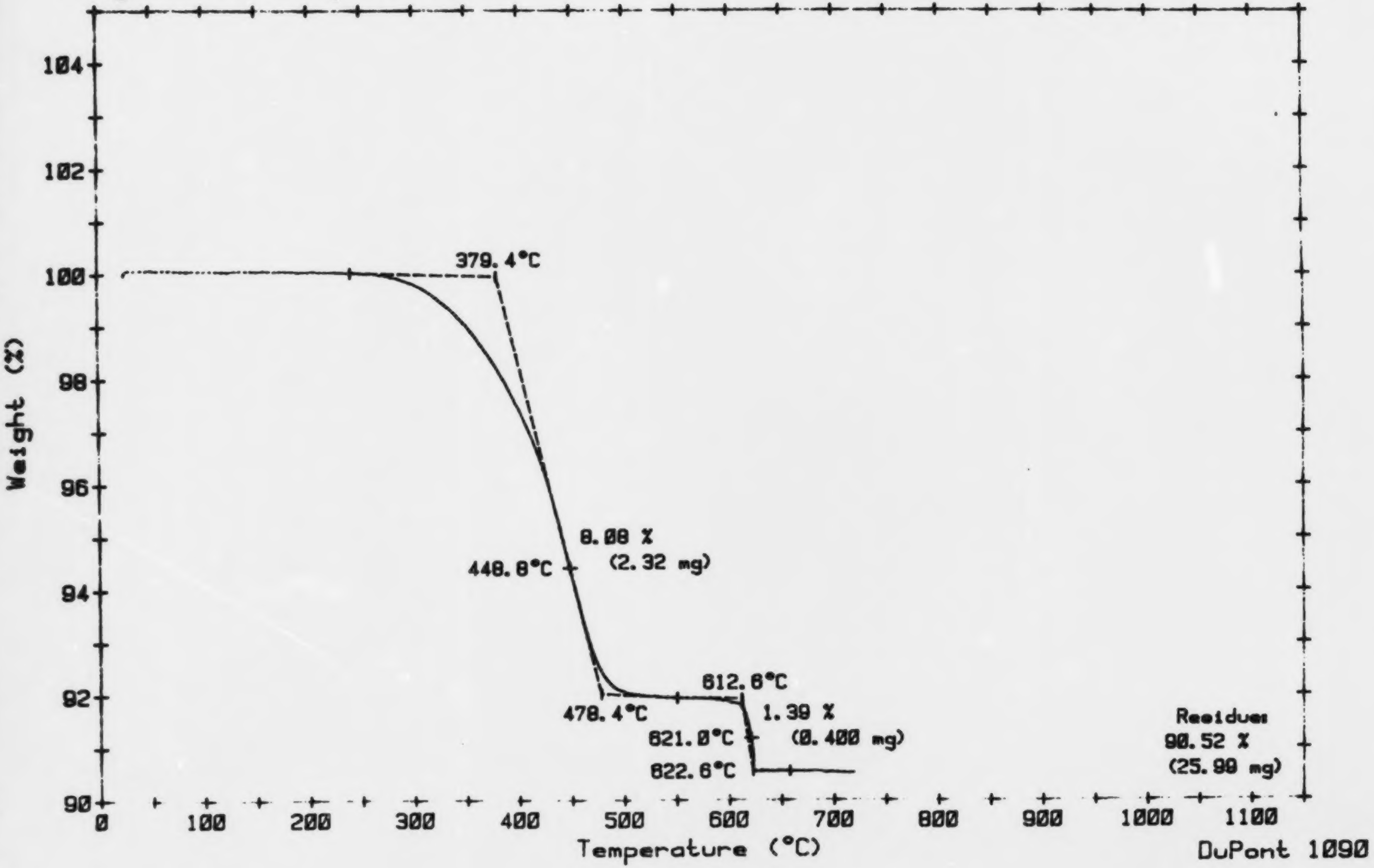
```
Sample: 10%MONT.80%SAND                                   Date: 22-Aug-85    Time:   8:36:05
Size: 26.47 mg                                            File: PEL-RESID.71 T.LYNCH.05
Rate: 10C/MIN                                             Operator: JT
Program: General Analysis V1.0                            Plotted: 23-Aug-85    14:53:32

      Temp        Sig-A      Deriv-A                  Temp       Sig-A      Deriv-A
      [deg]        [%]       [%/min]                  [deg]       [%]       [%/min]

      200.2      100.068      -0.02                   300.3      99.811      0.10
      205.5      100.076       0.02                   305.4      99.762      0.12
      210.1      100.068       0.00                   310.4      99.702      0.19
      215.3      100.072      -0.02                   315.5      99.626      0.13
      220.0      100.083      -0.01                   320.0      99.566      0.19
      225.3      100.079       0.01                   325.1      99.490      0.22
      230.4      100.072       0.05                   330.2      99.384      0.19
      235.0      100.072      -0.01                   335.2      99.290      0.21
      240.2      100.060       0.01                   340.3      99.184      0.24
      245.4      100.057       0.02                   345.4      99.071      0.27
      250.5      100.060       0.02                   350.4      98.961      0.29
      255.0      100.038       0.03                   355.4      98.844      0.29
      260.1      100.030       0.04                   360.5      98.704      0.31
      265.3      100.015       0.04                   365.0      98.584      0.36
      270.5      100.000       0.02                   370.5      98.391      0.34
      275.1       99.981       0.05                   375.5      98.236      0.36
      280.3       99.955       0.07                   380.5      98.081      0.34
      285.4       99.932       0.04                   385.4      97.907      0.38
      290.5       99.906       0.12                   390.4      97.734      0.41
      295.1       99.864       0.07                   395.3      97.533      0.45
                                                      400.3      97.326      0.52
```

```
Sample: 10%MONT.80%SAND                                 Date: 22-Aug-85    Time:  8:36:05
Size: 26.47 mg                                          File: PEL-RESID.71 T.LYNCH.05
Rate: 10C/MIN                                           Operator: JT
Program: General Analysis V1.0                          Plotted: 23-Aug-85   15:01:34

    Temp       Sig-A      Deriv-A                    Temp       Sig-A      Deriv-A
    [deg]       [%]       [%/min]                    [deg]       [%]       [%/min]

    400.3     97.326       0.52                      500.0     92.782       0.02
    405.2     97.107       0.49                      505.2     92.774       0.02
    410.2     96.869       0.57                      510.3     92.774       0.03
    415.1     96.616       0.58                      515.4     92.751       0.02
    420.5     96.302       0.66                      520.1     92.763       0.04
    425.4     95.981       0.73                      525.2     92.748      -0.01
    430.3     95.660       0.75                      530.3     92.736       0.02
    435.1     95.320       0.77                      535.5     92.721       0.01
    440.4     94.916       0.81                      540.0     92.725       0.00
    445.2     94.557       0.79                      545.1     92.710       0.00
    450.0     94.213       0.73                      550.1     92.710       0.04
    455.2     93.851       0.64                      555.1     92.710      -0.02
    460.5     93.545       0.57                      560.1     92.710       0.02
    465.3     93.318       0.43                      565.0     92.695       0.04
    470.0     93.148       0.32                      570.2     92.691       0.01
    475.3     93.016       0.21                      575.1     92.691       0.00
    480.0     92.921       0.14                      580.4     92.680       0.01
    485.3     92.861       0.10                      585.3     92.668       0.01
    490.1     92.838       0.06                      590.2     92.672      -0.01
    495.2     92.800       0.06                      595.1     92.657      -0.04
                                                     600.2     92.661       0.04
```

Sample: 10%MONT. 80%SAND
Size: 28.71 mg
Rate: 15C/MIN
Program: TGA Analysis V2.0

TGA

Date: 22-Aug-85 Time: 14:37:57
File: PEL-RESID.72 T.LYNCH.05
Operator: JT
Plotted: 22-Aug-85 15:36:10



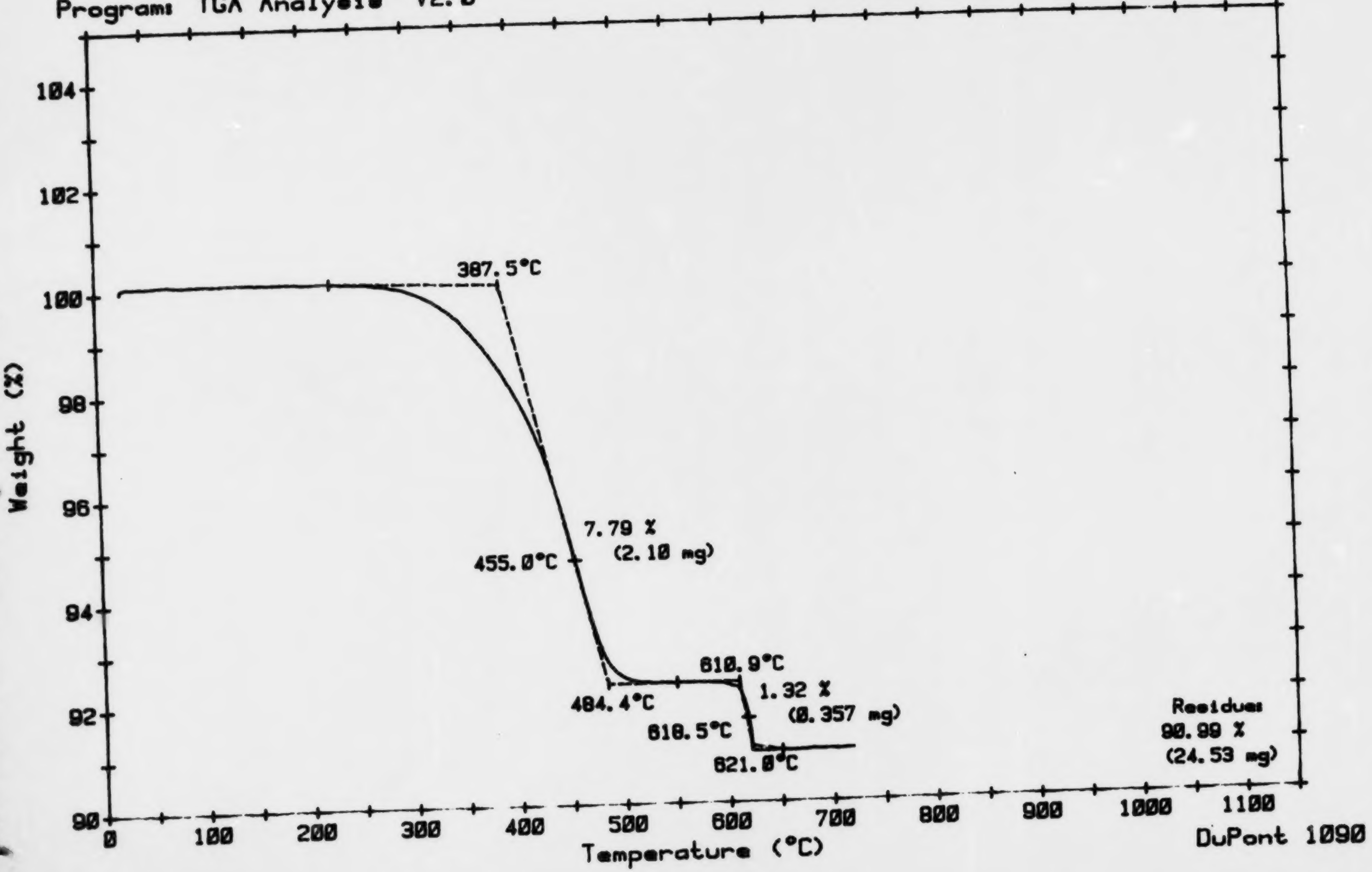
```
Sample: 10%MONT.80%SAND                          Date: 22-Aug-85    Time: 14:37:57
Size: 28.71 mg                                   File: PEL-RESID.72 T.LYNCH.05
Rate: 15C/MIN                                    Operator: JT
Program: General Analysis V1.0                   Plotted: 23-Aug-85    15:38:55

    Temp       Sig-A      Deriv-A                   Temp      Sig-A      Deriv-A
    [deg]       [%]       [%/min]                   [deg]      [%]       [%/min]

    200.6    100.049      -0.02                     300.1    99.812       0.15
    205.0    100.059      -0.02                     305.3    99.770       0.17
    210.4    100.049       0.02                     310.5    99.718       0.21
    215.7    100.052       0.00                     315.6    99.645       0.24
    220.1    100.049       0.00                     320.8    99.572       0.27
    225.4    100.045       0.01                     325.1    99.498       0.27
    230.7    100.042       0.03                     330.2    99.415       0.27
    235.1    100.042       0.00                     335.4    99.328       0.30
    240.3    100.028       0.00                     340.4    99.237       0.35
    245.5    100.038       0.03                     345.5    99.129       0.35
    250.7    100.031       0.02                     350.7    99.018       0.40
    255.1    100.021       0.01                     355.7    98.892       0.43
    260.3    100.014       0.03                     360.8    98.753       0.47
    265.5    100.010       0.07                     365.0    98.635       0.47
    270.7     99.979       0.03                     370.1    98.495       0.49
    275.1     99.976       0.08                     375.1    98.346       0.51
    280.3     99.937       0.07                     380.1    98.182       0.58
    285.5     99.927       0.10                     385.1    98.011       0.58
    290.6     99.885       0.12                     390.0    97.834       0.63
    295.7     99.843       0.12                     395.7    97.611       0.62
                                                    400.5    97.419       0.65
```

```
Sample: 10%MONT.80%SAND                                    Date: 22-Aug-85    Time: 14:37:57
Size: 28.71 mg                                             File: PEL-RESID.72 T.LYNCH.05
Rate: 15C/MIN                                              Operator: JT
Program: General Analysis V1.0                             Plotted: 23-Aug-85    15:47:16

     Temp        Sig-A        Deriv-A                  Temp       Sig-A       Deriv-A
     [deg]        [%]         [%/min]                  [deg]       [%]        [%/min]

     400.5       97.419        0.65                    500.3      92.062        0.10
     405.4       97.207        0.72                    505.7      92.024        0.06
     410.2       96.904        0.75                    510.3      92.013        0.06
     415.0       96.750        0.86                    515.0      91.992        0.03
     420.7       96.440        0.93                    520.3      91.988        0.03
     425.6       96.144        1.01                    525.7      91.985        0.01
     430.4       95.827        1.09                    530.3      91.982        0.02
     435.1       95.488        1.17                    535.5      91.968        0.03
     440.7       95.061        1.28                    540.7      91.961        0.02
     445.6       94.674        1.31                    545.2      91.964       -0.01
     450.4       94.208        1.26                    550.5      91.947        0.02
     455.1       93.905        1.32                    555.0      91.957        0.02
     460.7       93.458        1.19                    560.2      91.937        0.02
     465.5       93.121        1.06                    565.4      91.933        0.02
     470.4       92.825        0.91                    570.4      91.933       -0.01
     475.2       92.577        0.71                    575.1      91.930        0.01
     480.7       92.372        0.48                    580.3      91.919        0.01
     485.4       92.250        0.36                    585.5      91.919        0.06
     490.2       92.153        0.23                    590.6      91.908        0.02
     495.7       92.097        0.14                    595.1      91.891        0.04
                                                       600.1      91.874        0.10
```

Sample: 10%MONT.80%SAND
Size: 26.96 mg
Rate: 20C/MIN
Program: TGA Analysis V2.0

TGA

Date: 22-Aug-85 Time: 16:12:36
File: PEL-RESID.73 T.LYNCH.05
Operator: JT
Plotted: 23-Aug-85 7:47:54



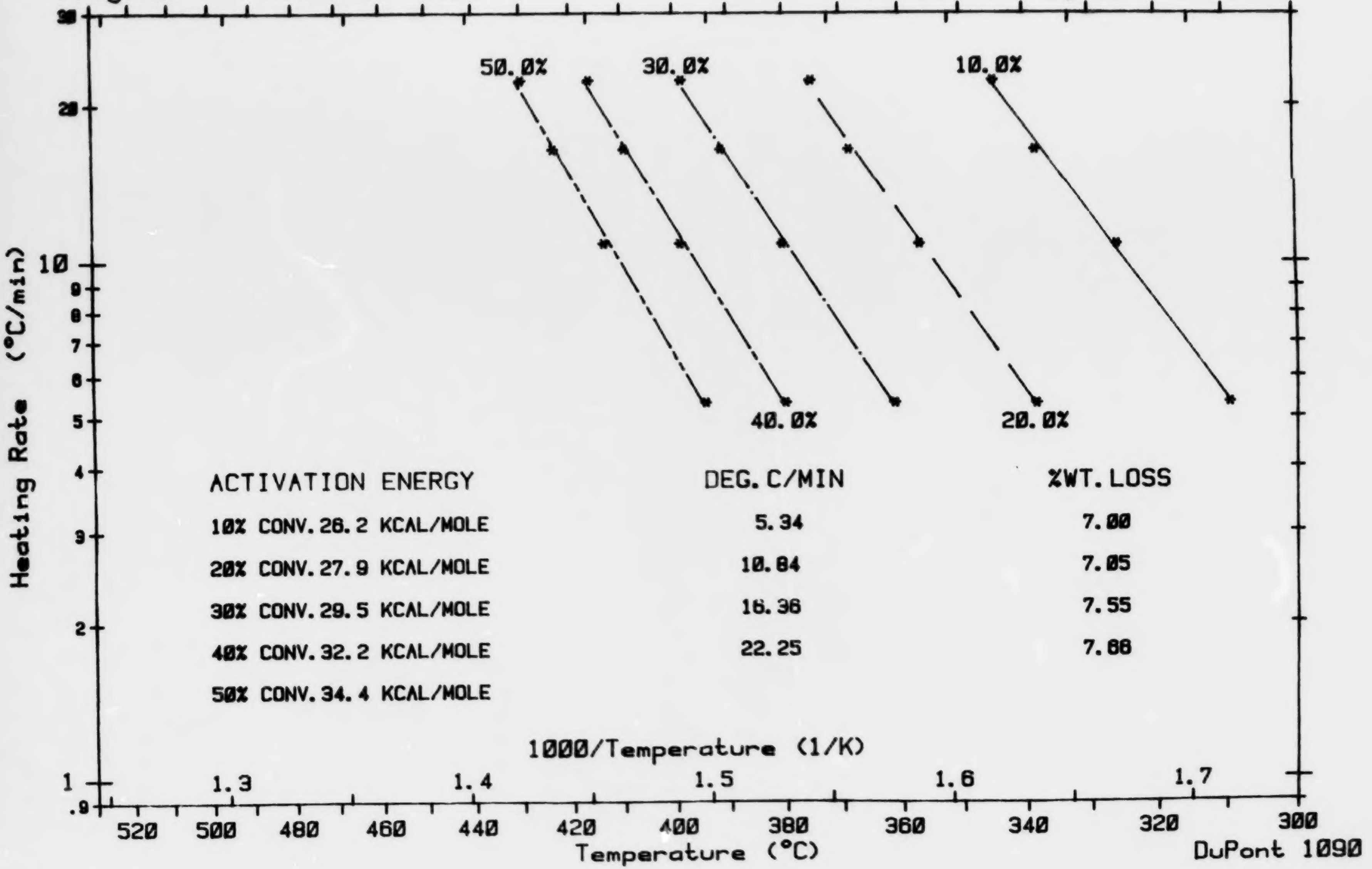
```
                                                    Date: 22-Aug-85    Time: 16:12:36
Sample: 10%MONT.80%SAND                             File: PEL-RESID.73 T.LYNCH.05
Size: 26.96 mg                                      Operator: JT
Rate: 20C/MIN                                       Plotted: 23-Aug-85    15:57:02
Program: General Analysis V1.0

     Temp       Sig-A      Deriv-A               Temp       Sig-A      Deriv-A
     [deg]       [%]      [%/min]                [deg]       [%]      [%/min]

     200.3     100.093       0.03                300.5     99.911       0.18
     205.0     100.100      -0.02                305.2     99.878       0.16
     211.0     100.100      -0.01                311.0     99.829       0.22
     215.7     100.100       0.00                315.6     99.781       0.23
     220.5     100.104       0.00                320.2     99.748       0.24
     225.3     100.100       0.00                325.9     99.674       0.29
     230.0     100.100       0.03                330.5     99.614       0.31
     235.8     100.082       0.04                335.1     99.548       0.33
     240.7     100.082       0.02                340.8     99.455       0.38
     245.4     100.078       0.02                345.3     99.384       0.38
     250.1     100.074       0.00                351.0     99.289       0.53
     256.0     100.074       0.02                355.5     99.182       0.50
     260.7     100.070       0.06                360.0     99.069       0.53
     265.4     100.052       0.07                365.6     98.924       0.53
     270.2     100.037       0.04                370.1     98.824       0.58
     276.0     100.026       0.07                375.7     98.689       0.64
     280.7     100.011       0.09                380.2     98.535       0.65
     285.4      99.989       0.11                385.8     98.372       0.67
     290.0      99.970       0.09                390.3     98.235       0.75
     295.9      99.948       0.14                395.9     98.042       0.78
                                                 400.3     97.879       0.81
```

```
Sample: 10%MONT.80%SAND                                     Date: 22-Aug-85    Time: 16:12:36
Size: 26.96 mg                                              File: PEL-RESID.73 T.LYNCH.05
Rate: 20C/MIN                                               Operator: JT
Program: General Analysis V1.0                              Plotted: 23-Aug-85    16:05:33

      Temp      Sig-A     Deriv-A                     Temp      Sig-A     Deriv-A
      [deg]     [%]       [%/min]                     [deg]     [%]       [%/min]

      400.3     97.879     0.81                       500.6     92.412     0.22
      405.9     97.671     0.86                       505.9     92.368     0.13
      410.2     97.500     0.92                       510.1     92.345     0.10
      415.8     97.248     1.01                       515.3     92.327     0.04
      420.1     97.040     1.08                       520.5     92.323     0.02
      425.7     96.759     1.20                       525.7     92.308     0.06
      430.0     96.506     1.33                       530.9     92.293    -0.01
      435.5     96.162     1.45                       535.1     92.316     0.00
      440.9     95.780     1.55                       540.2     92.305     0.00
      445.3     95.464     1.64                       545.4     92.301     0.01
      450.7     95.042     1.75                       550.5     92.308     0.00
      456.0     94.600     1.73                       555.7     92.297    -0.01
      460.3     94.252     1.72                       560.8     92.316     0.05
      465.6     93.833     1.61                       565.9     92.305     0.03
      470.9     93.454     1.47                       570.8     92.301     0.01
      475.2     93.172     1.25                       575.0     92.293     0.02
      480.5     92.887     1.00                       580.2     92.286     0.02
      485.8     92.687     0.65                       585.2     92.293     0.03
      490.0     92.579     0.48                       590.2     92.279     0.01
      495.3     92.483     0.33                       595.2     92.279     0.06
                                                      600.2     92.245     0.11
```

Sample: 45% MONTMORILLINITE　　Date: 24-Sep-85　　Time: 8:40:34

Operator: JT

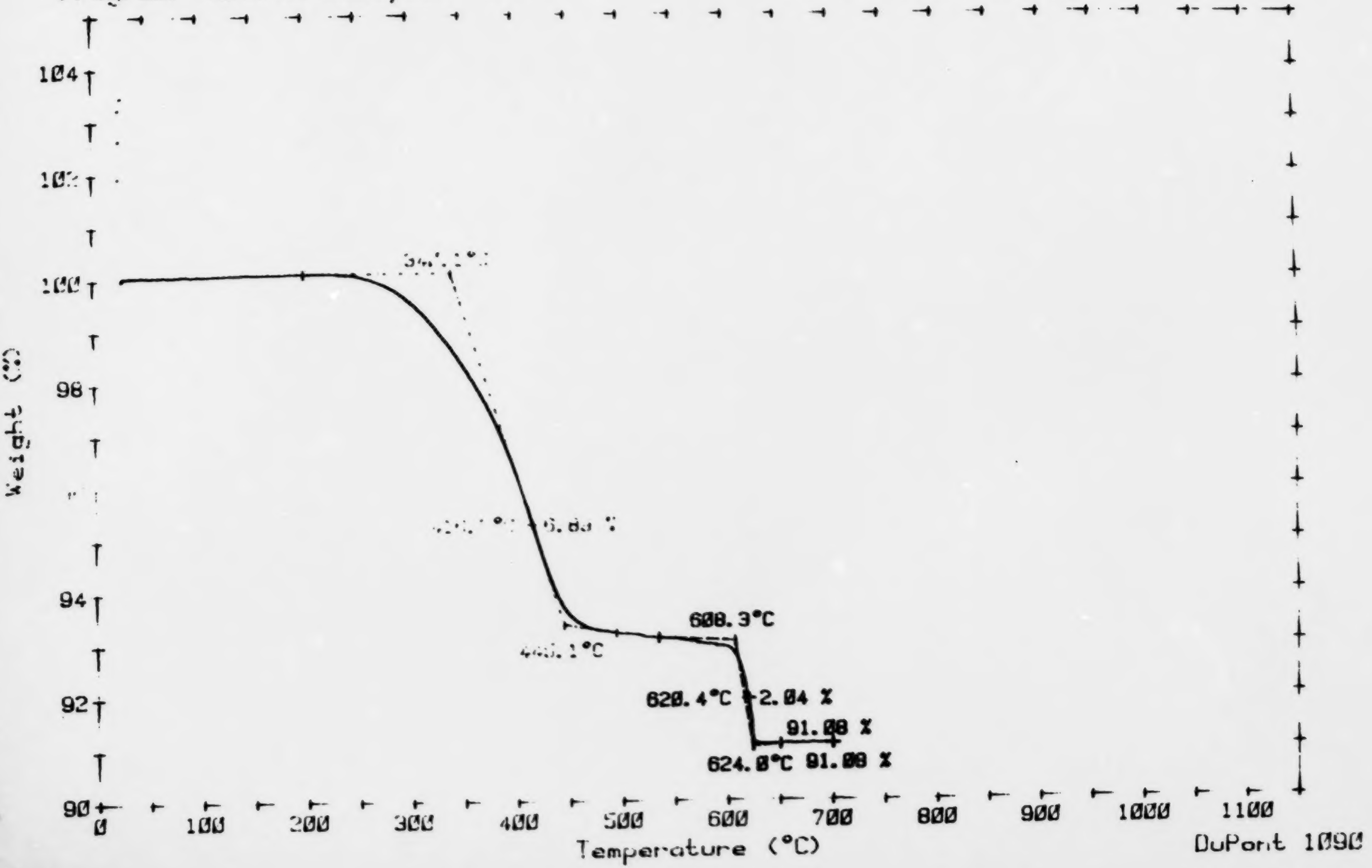
Program: TGA Kinetics　V1.0　　Plotted: 26-Sep-85　　15:33:30



ACTIVATION ENERGY	DEG. C/MIN	%WT. LOSS
10% CONV. 26.2 KCAL/MOLE	5.34	7.00
20% CONV. 27.9 KCAL/MOLE	10.84	7.05
30% CONV. 29.5 KCAL/MOLE	16.36	7.55
40% CONV. 32.2 KCAL/MOLE	22.25	7.66
50% CONV. 34.4 KCAL/MOLE		

Sample: 45% MONTMORILLONITE
Size: 28.89 mg
Rate: [illegible]
Program: General Analysis [illegible]

Date: 23-Sep-85 Time: [illegible]:23:36
File: PSL-RESIL.9M [illegible]
Operator: JT
Plotted: 30-Sep-85 8:34:[illegible]



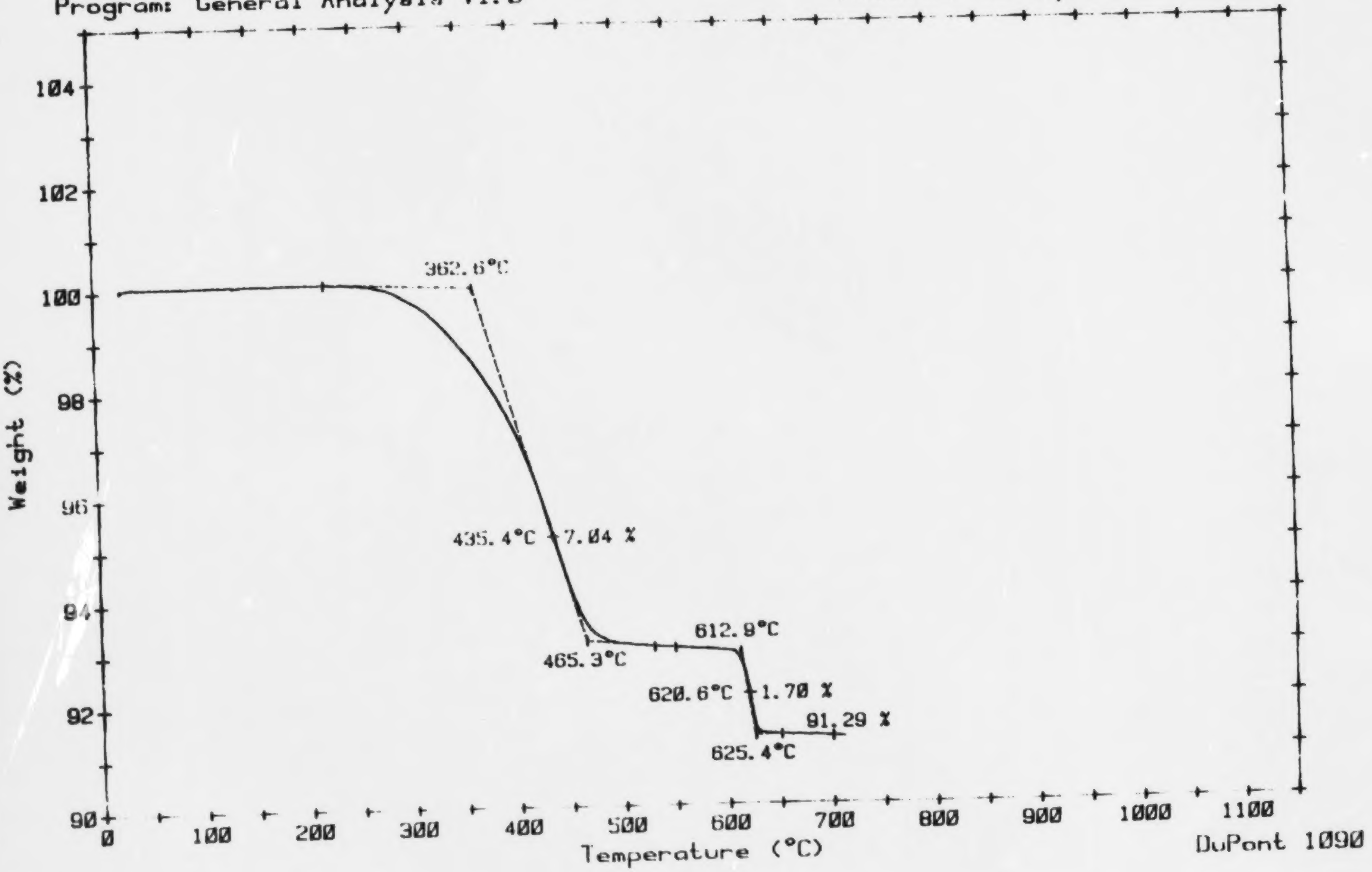
```
Sample: 45% MONTMORILLINITE                          Date: 23-Sep-85    Time: 16:23:36
Size: 28.89 mg                                       File: PEL-RESID.90 T.LYNCH.07
Rate: 05C/MIN                                        Operator: JT
Program: General Analysis V1.0                       Plotted: 30-Sep-85     8:49:16

     Temp       Sig-A      Deriv-A                       Temp       Sig-A      Deriv-A
     [deg]       [%]       [%/min]                       [deg]       [%]       [%/min]

     150.1     100.076       0.00                        250.2     100.038       0.01
     155.1     100.080      -0.01                        255.1     100.017       0.02
     160.0     100.080       0.00                        260.1      99.986       0.02
     165.1     100.087      -0.01                        265.1      99.958       0.02
     170.1     100.080       0.02                        270.0      99.920       0.01
     175.1     100.080       0.00                        275.2      99.875       0.02
     180.1     100.090      -0.01                        280.2      99.827       0.03
     185.1     100.090      -0.01                        285.1      99.778       0.03
     190.0     100.087       0.00                        290.2      99.709       0.05
     195.1     100.090       0.00                        295.1      99.637       0.04
     200.1     100.093       0.00                        300.1      99.547       0.03
     205.1     100.090       0.00                        305.0      99.463       0.06
     210.1     100.090       0.00                        310.2      99.360       0.06
     215.1     100.097      -0.01                        315.1      99.259       0.06
     220.1     100.090       0.00                        320.0      99.145       0.07
     225.0     100.087       0.01                        325.1      99.013       0.08
     230.2     100.087       0.02                        330.2      98.892       0.06
     235.1     100.069       0.00                        335.1      98.764       0.06
     240.0     100.062       0.00                        340.0      98.629       0.09
     245.2     100.059       0.00                        345.1      98.487       0.09
                                                         350.1      98.338       0.08
```

```
Sample: 45% MONTMORILLINITE                      Date: 23-Sep-85    Time: 16:23:36
Size: 28.89 mg                                   File: PEL-RESID.90 T.LYNCH.07
Rate: 05C/MIN                                    Operator: JT
Program: General Analysis V1.0                   Plotted: 30-Sep-85     8:57:46

    Temp       Sig-A      Deriv-A                Temp       Sig-A      Deriv-A
    [deg]       [%]       [%/min]                [deg]       [%]       [%/min]

    350.1     98.338        0.08                 450.0     93.565        0.06
    355.2     98.169        0.08                 455.1     93.468        0.05
    360.0     98.010        0.09                 460.2     93.395        0.03
    365.2     97.830        0.09                 465.1     93.340        0.02
    370.1     97.653        0.10                 470.0     93.302        0.02
    375.1     97.470        0.10                 475.1     93.274        0.00
    380.1     97.258        0.10                 480.1     93.250        0.01
    385.2     97.023        0.12                 485.2     93.236       -0.01
    390.0     96.798        0.13                 490.1     93.222        0.01
    395.0     96.538        0.14                 495.1     93.208        0.01
    400.1     96.258        0.16                 500.2     93.191       -0.01
    405.1     95.960        0.16                 505.1     93.194        0.00
    410.1     95.645        0.16                 510.2     93.187        0.00
    415.1     95.330        0.16                 515.2     93.181        0.01
    420.1     [illegible]   [illegible]          520.2     93.142        0.00
    425.1     94.683        0.16                 525.1     93.125        0.02
    430.0     94.389        0.14                 530.1     93.108        0.00
    435.2     94.112        0.14                 535.0     93.104        0.00
    440.1     93.880        0.09                 540.1     93.094        0.01
    445.0     93.703        0.07                 545.0     93.084        0.00
                                                 550.2     93.077        0.02
```

Sample: 45% MONTMORILLONITE
Size: 28.53 mg
Rate: 10C/MIN
Program: General Analysis V1.0

TGA

Date: 26-Sep-85 Time: 13:46:29
File: PEL-RESID.91 T.LYNCH.07
Operator: JT
Plotted: 30-Sep-85 9:32:11



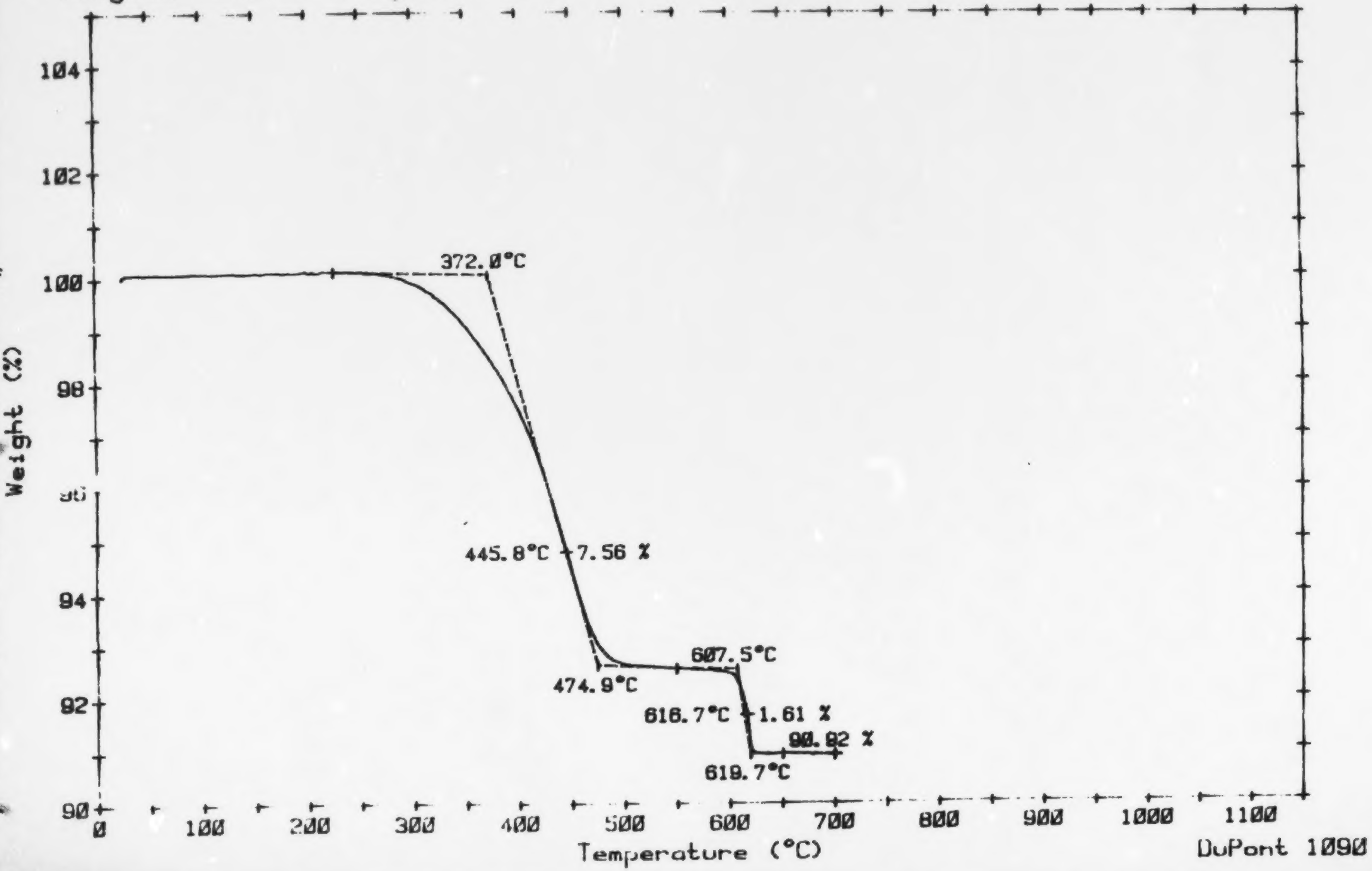
```
Sample: 45% MONTMORILLONITE                                    Date: 26-Sep-85     Time: 13:46:29
Size: 28.53 mg                                                 File: PEL-RESID.91 T.LYNCH.07
Rate: 10C/MIN                                                  Operator: JT
Program: General Analysis V1.0                                 Plotted: 30-Sep-85     9:43:57

      Temp        Sig-A       Deriv-A                        Temp        Sig-A       Deriv-A
      [deg]        [%]        [%/min]                        [deg]        [%]        [%/min]

      150.1      100.081        0.01                         250.1      100.070        0.01
      155.2      100.084        0.00                         255.3      100.053        0.03
      160.5      100.088        0.00                         260.2      100.042        0.01
      165.1      100.081       -0.01                         265.2      100.035        0.02
      170.1      100.077        0.00                         270.3      100.007        0.02
      175.3      100.091        0.00                         275.2       99.982        0.02
      180.5      100.091        0.00                         280.2       99.961        0.03
      185.2      100.095        0.00                         285.3       99.923        0.04
      190.3      100.091        0.00                         290.3       99.870        0.07
      195.4      100.088        0.00                         295.3       99.804        0.06
      200.4      100.091       -0.01                         300.3       99.772        0.06
      205.5      100.088        0.00                         305.2       99.709        0.06
      210.2      100.084        0.01                         310.0       99.649        0.07
      215.2      100.088       -0.01                         315.0       99.572        0.08
      220.4     [illegible]   [illegible]                    320.0       99.485        0.09
      225.5      100.084        0.00                         325.3       99.394        0.10
      230.1      100.077        0.01                         330.2       99.295        0.12
      235.1      100.074        0.00                         335.2       99.190        0.13
      240.1      100.074        0.02                         340.1       99.075        0.13
      245.1      100.063        0.01                         345.4       98.952        0.12
                                                             350.4       98.833        0.14
```

```
Sample:  45% MONTMORILLONITE                       Date:  26-Sep-85    Time:  13:46:29
Size:  28.53 mg                                    File:  PEL-RESID.91  T.LYNCH.07
Rate:  10C/MIN                                     Operator:  JT
Program:  General Analysis V1.0                    Plotted:  30-Sep-85     9:58:11

      Temp        Sig-A       Deriv-A                   Temp        Sig-A       Deriv-A
      [deg]        [%]        [%/min]                   [deg]        [%]        [%/min]

      350.4       98.833        0.14                    450.2       94.234        0.32
      355.3       98.717        0.12                    455.4       93.936        0.28
      360.1       98.587        0.15                    460.1       93.712        0.23
      365.1       98.447        0.15                    465.4       93.522        0.17
      370.5       98.286        0.16                    470.1       93.396        0.12
      375.4       98.125        0.18                    475.3       93.295        0.07
      380.3       97.967        0.18                    480.0       93.238        0.04
      385.2       97.799        0.21                    485.2       93.186        0.05
      390.0       97.620        0.20                    490.4       93.161        0.04
      395.4       97.406        0.22                    495.0       93.133        0.02
      400.3       97.206        0.23                    500.3       93.119        0.01
      405.0       96.986        0.26                    505.3       93.109        0.01
      410.4       96.719        0.28                    510.4       93.098        0.02
      415.2       96.467        0.30                    515.0       93.077        0.01
      420.[illegible]      [illegible]   [illegible]      520.0       93.070        0.01
      425.4       95.864        0.33                    525.1       93.070        0.01
      430.2       95.555        0.35                    530.2       93.056       -0.01
      435.4       95.187        0.35                    535.3       93.046        0.02
      440.1       94.879        0.35                    540.3       93.039        0.00
      445.5       94.539        0.34                    545.3       93.039        0.00
                                                        550.3       93.032        0.01
```

Sample: 45% MONTMORILLINITE
Size: 27.44 mg
Rate: 15C/MIN
Program: General Analysis V1.0

TGA

Date: 24-Sep-85 Time: 10:14:19
File: PEL-RESID.93 T.LYNCH.07
Operator: JT
Plotted: 30-Sep-85 10:02:59



DuPont 1090

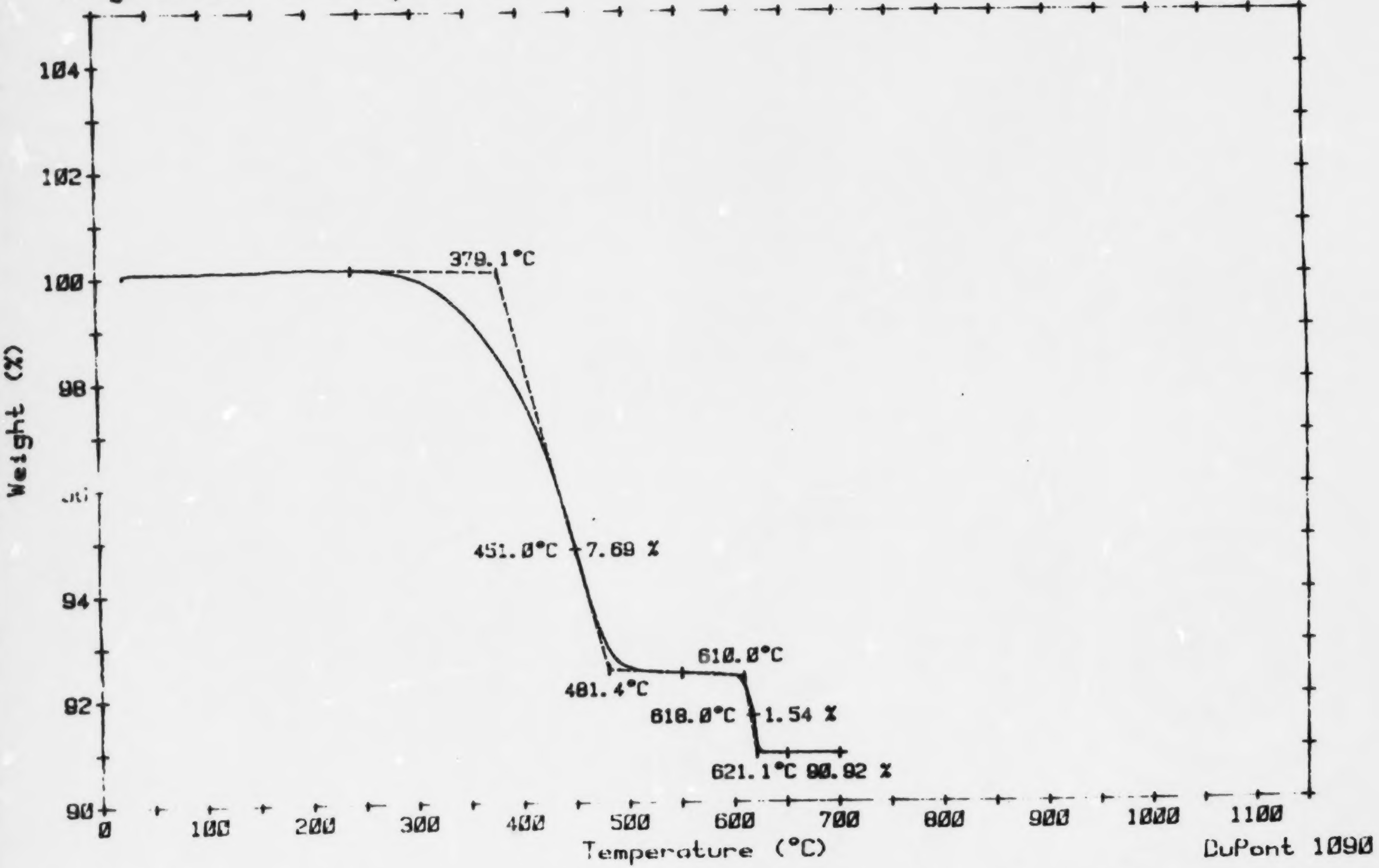
```
Sample: 45% MONTMORILLINITE                          Date: 24-Sep-85   Time: 10:14:19
Size: 27.44 mg                                       File: PEL-RESID.93 T.LYNCH.07
Rate: 15C/MIN                                        Operator: JT
Program: General Analysis V1.0                       Plotted: 30-Sep-85   10:18:17

    Temp      Sig-A      Deriv-A                       Temp      Sig-A      Deriv-A
    [deg]      [%]       [%/min]                       [deg]      [%]       [%/min]

    150.5    100.098      -0.01                        250.7    100.106      0.00
    155.8    100.091       0.01                        255.7    100.087      0.01
    160.2    100.095      -0.03                        260.7    100.080      0.00
    165.4    100.106       0.00                        265.8    100.091      0.02
    170.6    100.098       0.00                        270.7    100.084      0.03
    175.8    100.106       0.00                        275.0    100.062      0.04
    180.0    100.098       0.00                        280.0    100.029      0.04
    185.1    100.102       0.00                        285.1    100.011      0.05
    190.3    100.109      -0.01                        290.2     99.978      0.04
    195.5    100.106      -0.03                        295.3     99.949      0.08
    200.6    100.120       0.02                        300.2     99.898      0.07
    205.7    100.106      -0.02                        305.3     99.851      0.08
    210.8    100.117       0.01                        310.3     99.800      0.10
    215.2    100.128      -0.03                        315.2     99.723      0.10
    220.4    100.124      [illegible]                  320.2     99.676      0.15
    225.4    100.117       0.00                        325.2     99.588      0.15
    230.5    100.120       0.00                        330.2     99.504      0.14
    235.6    100.117      -0.01                        335.1     99.402      0.17
    240.7    100.124       0.02                        340.1     99.315      0.17
    245.6    100.124       0.00                        345.0     99.202      0.20
                                                       350.0     99.078      0.20
```

```
Sample:  45% MONTMORILLINITE                    Date:  24-Sep-85    Time:  10:14:19
Size:  27.44 mg                                 File:  PEL-RESID.93 T.LYNCH.07
Rate:  15C/MIN                                  Operator:  JT
Program:  General Analysis V1.0                 Plotted:  30-Sep-85    10:47:12

    Temp        Sig-A       Deriv-A                  Temp        Sig-A       Deriv-A
    [deg]        [%]       [%/min]                   [deg]        [%]       [%/min]

    350.0      99.078         0.20                   450.6      94.403         0.58
    355.8      98.936         0.22                   455.3      94.068         0.55
    360.8      98.801         0.23                   460.8      93.704         0.49
    365.7      98.659         0.23                   465.5      93.427         0.41
    370.7      98.521         0.24                   470.3      93.201         0.34
    375.6      98.382         0.25                   475.7      92.983         0.24
    380.5      98.229         0.26                   480.4      92.865         0.18
    385.4      98.058         0.29                   485.7      92.763         0.12
    390.3      97.872         0.31                   490.4      92.709         0.08
    395.2      97.686         0.32                   495.0      92.665         0.05
    400.1      97.489         0.33                   500.4      92.636         0.04
    405.8      97.249         0.36                   505.7      92.625         0.02
    410.6      97.016         0.39                   510.3      92.603         0.02
    415.5      96.768         0.44                   515.6      92.599         0.01
    420.2      [illegible]    [illegible]            520.1      92.589         0.01
    425.1      96.214         0.50                   525.3      92.585         0.01
    430.7      95.842         0.55                   530.7      92.570         0.02
    435.4      95.496         0.57                   535.2      92.567         0.02
    440.1      95.150         0.57                   540.4      92.559         0.00
    445.0      94.808         0.58                   545.6      92.552         0.00
                                                     550.1      92.552         0.01
```

Sample: 45% MONTMORILLINITE
Size: 25.59 mg
Rate: 20C/MIN
Program: General Analysis V1.0

TGA

Date: 24-Sep-85 Time: 8:40:34
File: PEL-RESID.94 T.LYNCH.07
Operator: JT
Plotted: 30-Sep-85 10:51:38



DuPont 1090

```
Sample: 45% MONTMORILLINITE                     Date: 24-Sep-85    Time:   8:40:34
Size: 25.59 mg                                  File: PEL-RESID.94 T.LYNCH.07
Rate: 20C/MIN                                   Operator: JT
Program: General Analysis V1.0                  Plotted: 30-Sep-85    11:03:25

     Temp       Sig-A      Deriv-A                   Temp      Sig-A      Deriv-A
     [deg]       [%]       [%/min]                   [deg]      [%]       [%/min]

     151.1     100.121       0.01                    250.8    100.145       0.02
     155.7     100.117       0.00                    255.4    100.141       0.01
     160.4     100.121      -0.02                    261.1    100.133       0.01
     165.1     100.137      -0.03                    265.6    100.125       0.03
     170.8     100.145      -0.01                    270.2    100.109       0.04
     175.4     100.149       0.00                    275.9    100.094       0.04
     180.1     100.145      -0.01                    280.5    100.078       0.04
     185.9     100.152       0.01                    285.2    100.055       0.06
     190.6     100.152       0.00                    291.0    100.027       0.07
     195.2     100.152      -0.01                    295.5     99.996       0.08
     201.0     100.168      -0.01                    300.1     99.961       0.09
     205.7     100.160       0.01                    305.8     99.922       0.09
     210.3     100.152       0.01                    310.3     99.879       0.11
     216.1     100.152       0.01                    315.9     99.812       0.13
     220.8     100.[illegible]    [illegible]        320.5     99.754       0.15
     225.5     100.156       0.01                    325.1     99.695       0.17
     230.1     100.152      -0.01                    330.7     99.609       0.19
     235.8     100.156       0.01                    335.2     99.523       0.21
     240.4     100.149       0.00                    340.9     99.422       0.21
     245.1     100.152       0.01                    345.5     99.332       0.23
                                                     350.1     99.238       0.25
```

```
Sample: 45% MONTMORILLINITE                          Date: 24-Sep-85    Time:  8:40:34
Size: 25.59 mg                                       File: PEL-RESID.94 T.LYNCH.07
Rate: 20C/MIN                                        Operator: JT
Program: General Analysis V1.0                       Plotted: 30-Sep-85    11:12:04

      Temp       Sig-A      Deriv-A                       Temp       Sig-A      Deriv-A
      [deg]       [%]       [%/min]                       [deg]       [%]       [%/min]

      350.1      99.238       0.25                        451.0      94.821       0.80
      355.7      99.121       0.27                        455.2      94.497       0.80
      360.2      98.995       0.30                        460.7      94.102       0.77
      365.7      98.847       0.31                        465.0      93.801       0.72
      370.2      98.726       0.31                        470.3      93.453       0.61
      375.7      98.562       0.32                        475.5      93.187       0.50
      380.1      98.437       0.34                        480.8      92.957       0.40
      385.7      98.265       0.36                        485.0      92.816       0.32
      390.1      98.108       0.38                        490.3      92.691       0.19
      395.6      97.917       0.40                        495.6      92.621       0.12
      400.1      97.756       0.43                        500.8      92.570       0.08
      405.6      97.534       0.47                        506.0      92.539       0.05
      410.0      97.330       0.50                        510.2      92.523       0.05
      415.4      97.092       0.52                        515.3      92.503       0.02
      420.9      [illegible]  [illegible]                 520.4      92.492       0.02
      425.2      96.564       0.62                        525.5      92.480       0.01
      430.6      96.240       0.68                        530.6      92.468       0.01
      435.9      95.892       0.70                        535.6      92.472      -0.01
      440.2      95.607       0.74                        540.8      92.468       0.01
      445.6      95.216       0.80                        545.8      92.472       0.00
                                                          550.9      92.460       0.01
```

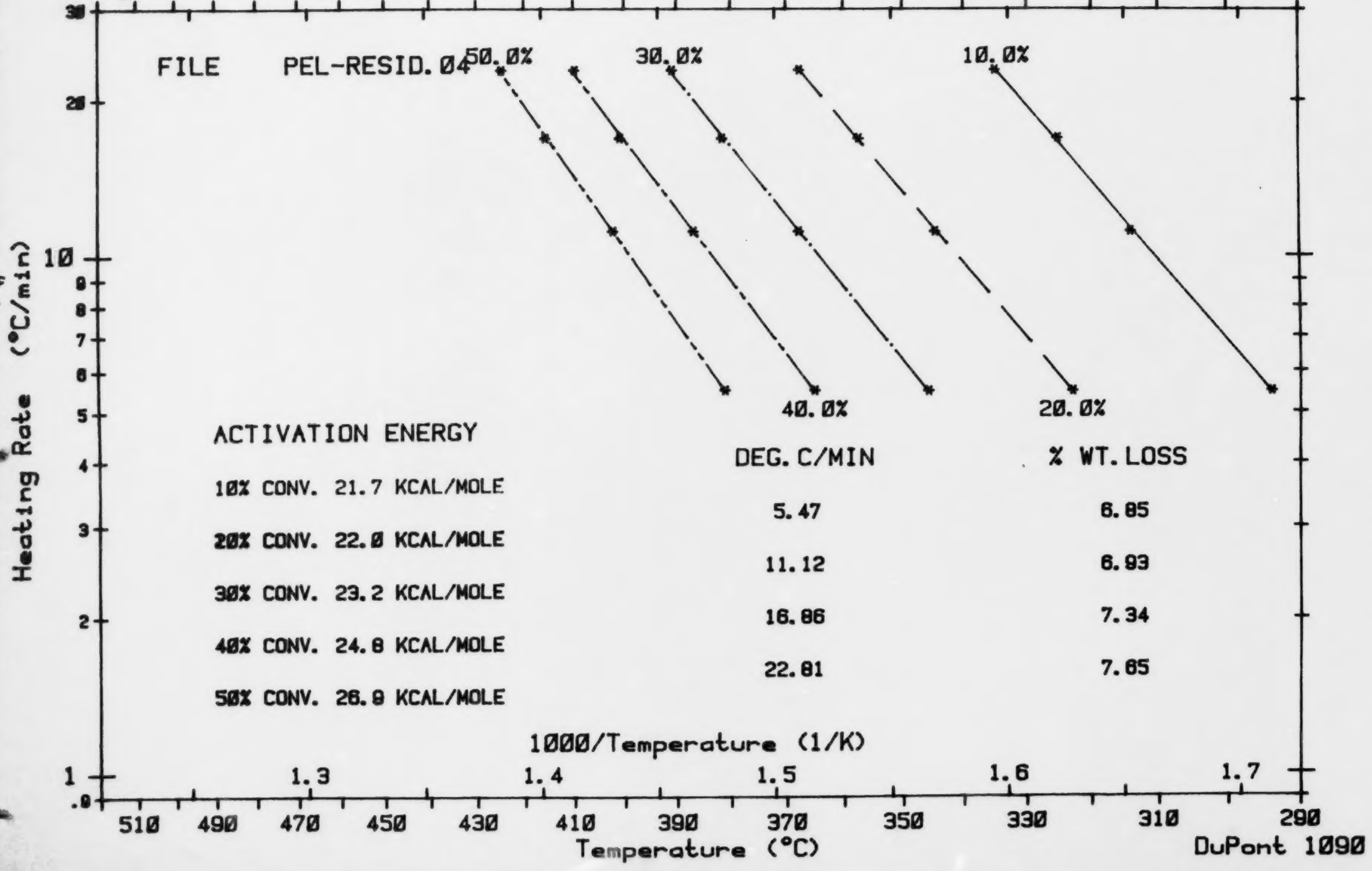
Sample: 90% MONTMORILLONITE

Date: 11-Jul-85 Time: 10:38:53

Operator: JT

Program: TGA Kinetics V1.0

Plotted: 18-Jul-85 16:29:36



ACTIVATION ENERGY

10% CONV. 21.7 KCAL/MOLE

20% CONV. 22.0 KCAL/MOLE

30% CONV. 23.2 KCAL/MOLE

40% CONV. 24.8 KCAL/MOLE

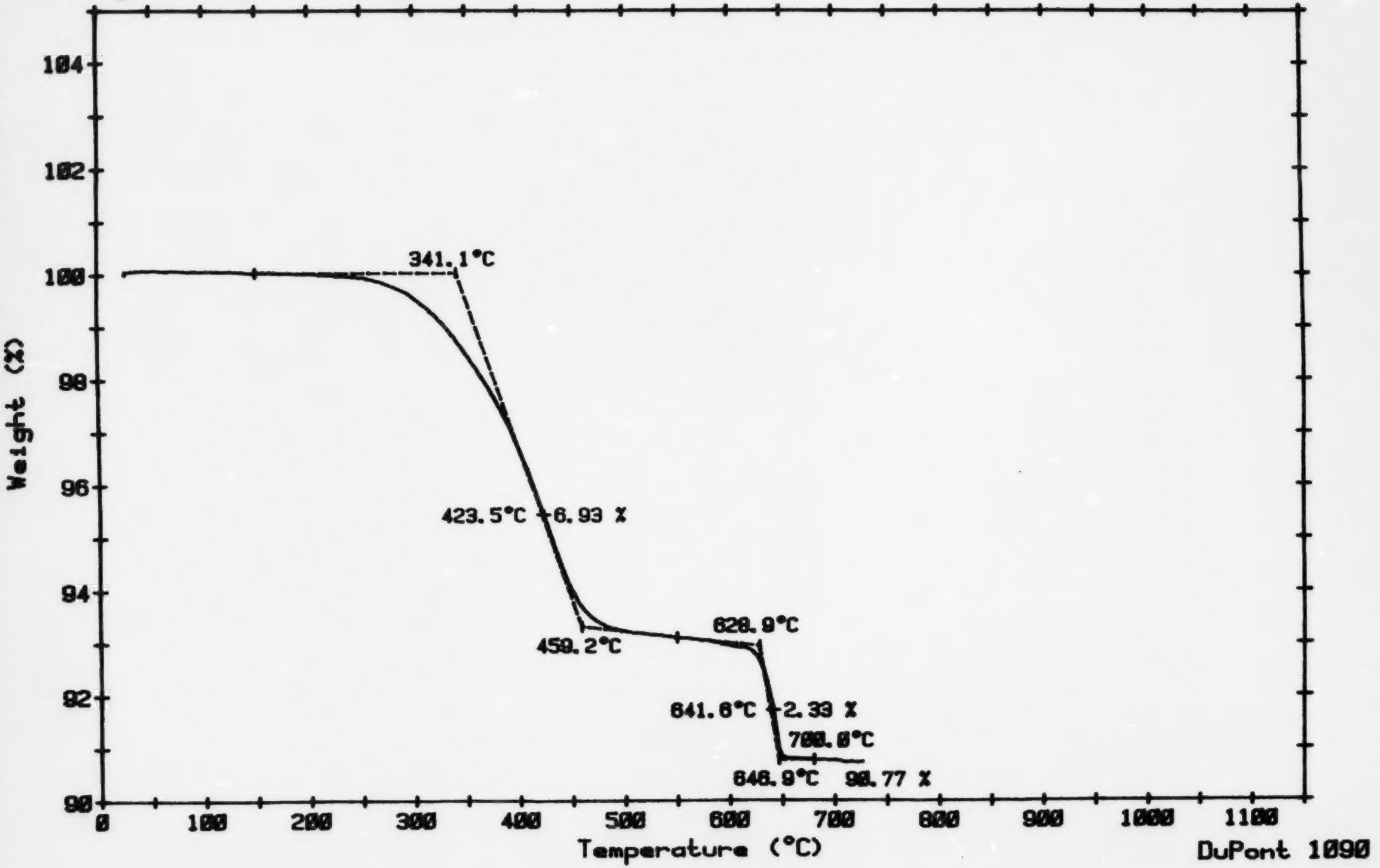
50% CONV. 26.9 KCAL/MOLE

DEG. C/MIN	% WT. LOSS
5.47	6.85
11.12	6.93
16.86	7.34
22.81	7.65

Sample: 90% MONTMORILLONITE
Size: 27.02 mg
Rate: 10C/MIN
Program: General Analysis V1.0

TGA

Date: 11-Jul-85 Time: 15:37:24
File: PEL-RESID.40 T.LYNCH.03
Operator: JT
Plotted: 18-Jul-85 14:34:13



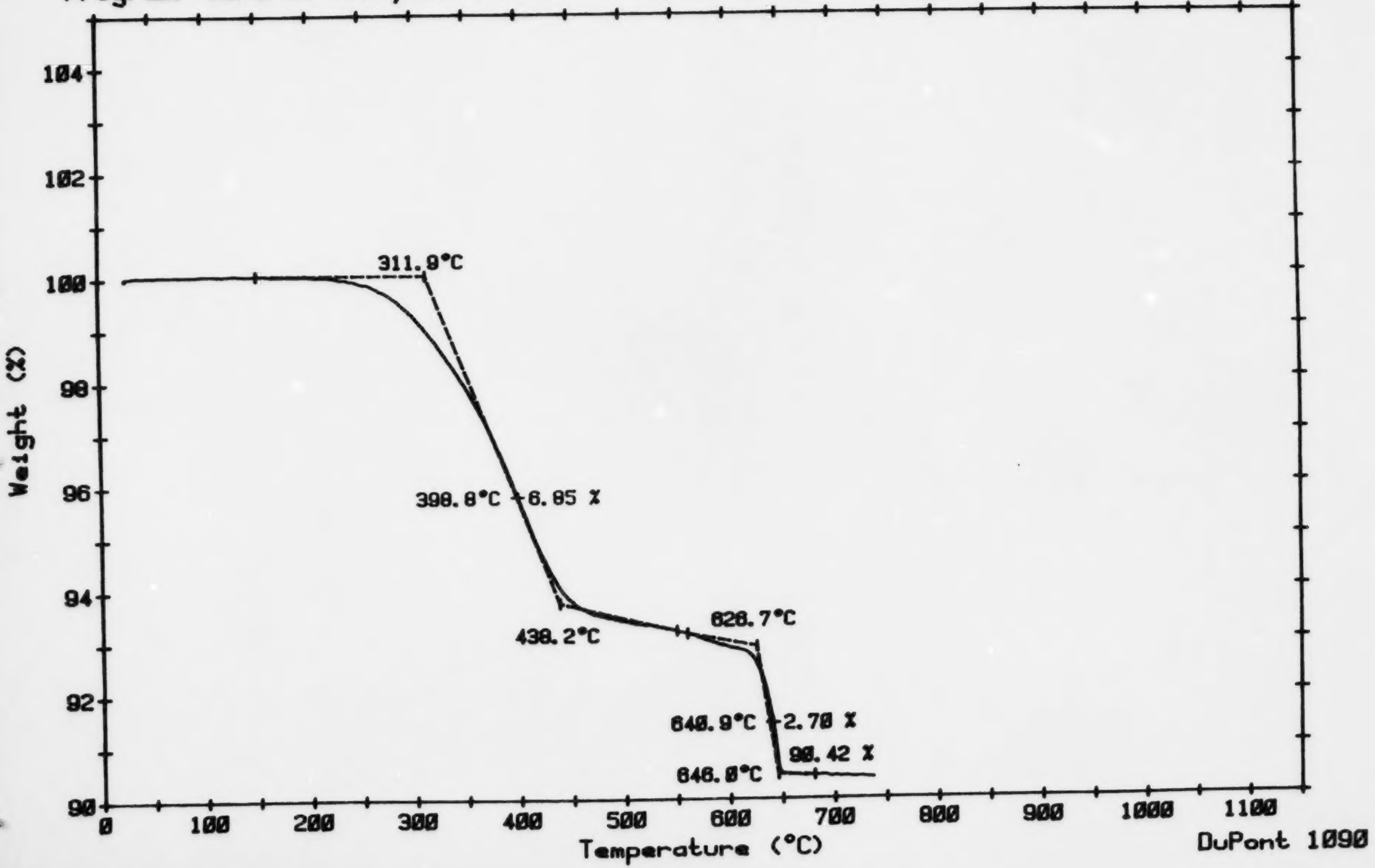
```
Sample: 90% MONTMORILLONITE                              Date: 11-Jul-85    Time: 15:37:24
Size: 27.02 mg                                           File: PEL-RESID.40 T.LYNCH.03
Rate: 10C/MIN                                            Operator: JT
Program: General Analysis V1.0                           Plotted: 18-Jul-85    14:55:22

     Temp        Sig-A      Deriv-A                  Temp        Sig-A      Deriv-A
    [deg]         [%]       [%/min]                 [deg]         [%]       [%/min]

    150.1      100.044       -0.02                  250.5       99.952        0.08
    155.5      100.052       -0.02                  255.2       99.944        0.09
    160.2      100.048       -0.01                  260.4       99.922        0.07
    165.6      100.056       -0.03                  265.0       99.889        0.07
    170.3      100.052        0.03                  270.2       99.870        0.08
    175.1      100.044        0.00                  275.5       99.822        0.09
    180.4      100.033        0.01                  280.1       99.785        0.09
    185.2      100.030       -0.01                  285.3       99.741        0.10
    190.5      100.026        0.04                  290.5       99.697        0.11
    195.3      100.026        0.01                  295.2       99.645        0.15
    200.0      100.033        0.01                  300.3       99.571        0.18
    205.3      100.026       -0.01                  305.5       99.489        0.18
    210.0      100.015        0.01                  310.1       99.415        0.18
    215.3      100.015        0.02                  315.3       99.326        0.24
    220.1      100.011        0.03                  320.5       99.234        0.23
    225.4       99.996        0.04                  325.0       99.127        0.23
    230.1       99.985       -0.01                  330.2       99.023        0.28
    235.4       99.965        0.06                  335.3       98.906        0.29
    240.0       99.974       -0.01                  340.4       98.768        0.34
    245.3       99.967        0.01                  345.4       98.627        0.32
                                                    350.4       98.486        0.31
```

```
Sample: 90% MONTMORILLONITE                    Date: 11-Jul-85    Time: 15:37:24
Size: 27.02 mg                                 File: PEL-RESID.40 T.LYNCH.03
Rate: 10C/MIN                                  Operator: JT
Program: General Analysis V1.0                 Plotted: 18-Jul-85    15:04:06

      Temp       Sig-A      Deriv-A                  Temp       Sig-A      Deriv-A
      [deg]       [%]       [%/min]                  [deg]       [%]       [%/min]

      350.4      98.486       0.31                   450.0      94.008       0.36
      355.5      98.335       0.35                   455.1      93.823       0.40
      360.0      98.209       0.35                   460.3      93.672       0.25
      365.0      98.053       0.33                   465.0      93.579       0.21
      370.0      97.887       0.37                   470.2      93.475       0.18
      375.1      97.709       0.39                   475.3      93.409       0.14
      380.1      97.532       0.41                   480.4      93.346       0.11
      385.2      97.339       0.43                   485.0      93.313       0.08
      390.2      97.139       0.46                   490.1      93.287       0.05
      395.1      96.917       0.51                   495.3      93.264       0.03
      400.1      96.681       0.53                   500.4      93.246       0.03
      405.0      96.444       0.53                   505.1      93.224       0.03
      410.4      96.173       0.58                   510.3      93.202      -0.03
      415.3      95.899       0.60                   515.0      93.205       0.03
      420.2      95.622       0.62                   520.1      93.194       0.05
      425.1      95.341       0.61                   525.1      93.165       0.02
      430.4      95.020       0.60                   530.3      93.165       0.00
      435.2      94.756       0.58                   535.2      93.142       0.03
      440.0      94.493       0.56                   540.2      93.146      -0.02
      445.3      94.223       0.54                   545.2      93.124       0.05
                                                     550.2      93.113       0.02
```

Sample: 90% MONTMORILLONITE
Size: 27.07 mg
Rate: 5C/MIN
Program: General Analysis V1.0

TGA

Date: 11-Jul-85 Time: 17:10:25
File: PEL-RESID.41 T.LYNCH.03
Operator: JT
Plotted: 18-Jul-85 15:09:54



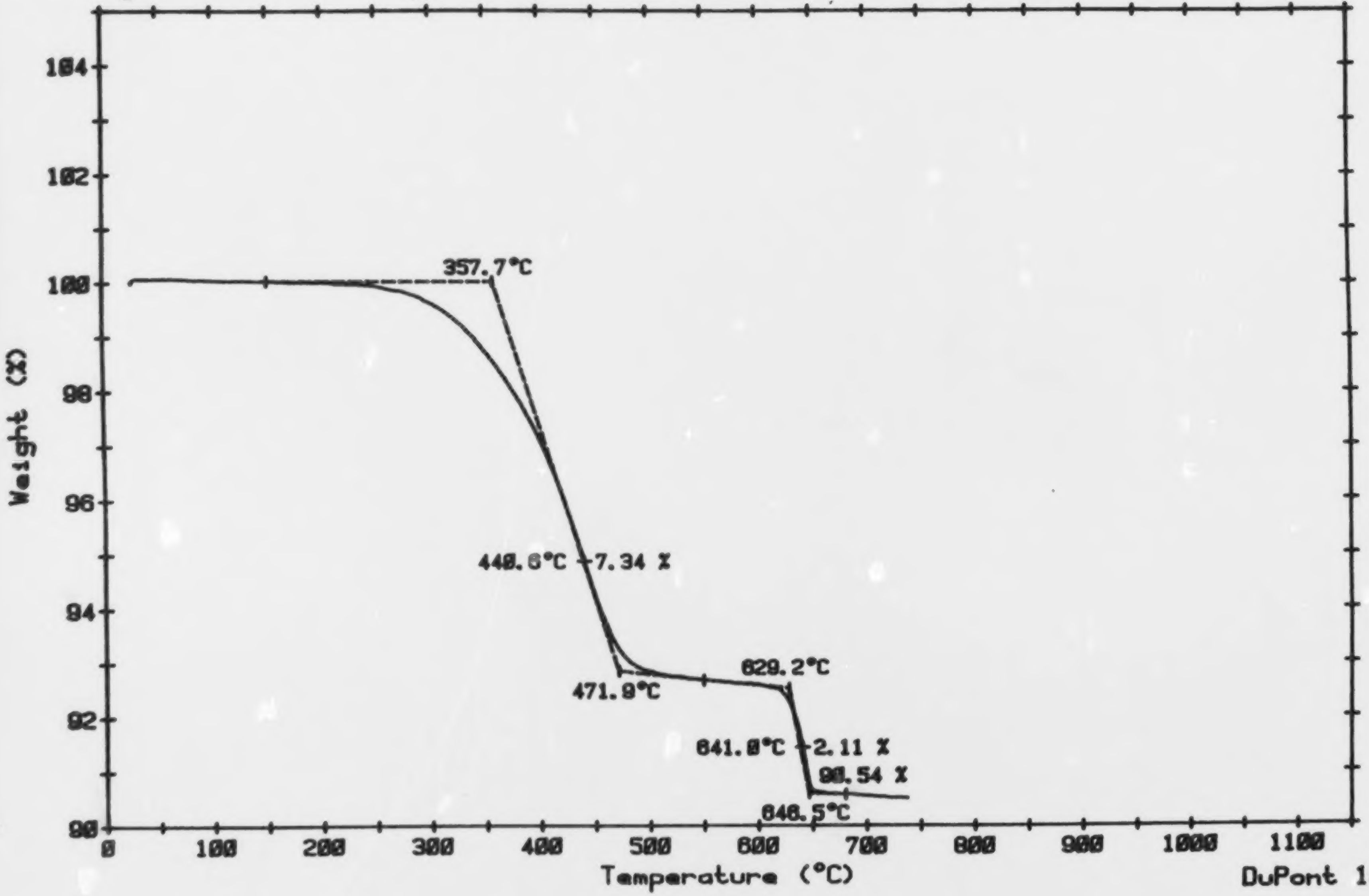
```
Sample: 90% MONTMORILLONITE                      Date: 11-Jul-85    Time: 17:10:25
Size: 27.07 mg                                   File: PEL-RESID.41 T.LYNCH.03
Rate:  5C/MIN                                    Operator: JT
Program: General Analysis V1.0                   Plotted: 18-Jul-85    15:20:50

     Temp      Sig-A      Deriv-A                    Temp      Sig-A      Deriv-A
     [deg]      [%]       [%/min]                    [deg]      [%]       [%/min]

     150.2    100.044      -0.02                     250.0     99.871       0.04
     155.3    100.041       0.01                     255.2     99.849       0.02
     160.0    100.037       0.01                     260.2     99.815       0.03
     165.0    100.030      -0.01                     265.1     99.764       0.05
     170.1    100.030      -0.03                     270.1     99.719       0.06
     175.1    100.026       0.03                     275.2     99.660       0.08
     180.1    100.022       0.02                     280.1     99.590       0.08
     185.1    100.015       0.02                     285.2     99.523       0.09
     190.2    100.022       0.00                     290.2     99.427       0.08
     195.2    100.033       0.02                     295.2     99.346       0.11
     200.1    100.018       0.01                     300.3     99.235       0.15
     205.1    100.007       0.01                     305.2     99.121       0.13
     210.1    100.007       0.01                     310.1     99.006       0.17
     215.1    100.000      -0.01                     315.2     98.881       0.15
     220.1     99.993       0.00                     320.2     98.750       0.14
     225.0     99.974       0.00                     325.1     98.611       0.14
     230.2     99.963       0.00                     330.0     98.470       0.16
     235.1     99.948       0.00                     335.0     98.323       0.15
     240.0     99.933       0.02                     340.0     98.164       0.17
     245.2     99.900       0.06                     345.0     98.020       0.16
                                                     350.0     97.848       0.22
```

```
Sample: 90% MONTMORILLONITE                      Date: 11-Jul-85    Time: 17:10:25
Size: 27.07 mg                                   File: PEL-RESID.41 T.LYNCH.03
Rate:  5C/MIN                                    Operator: JT
Program: General Analysis V1.0                   Plotted: 18-Jul-85    15:28:58

      Temp      Sig-A     Deriv-A              Temp      Sig-A     Deriv-A
     [deg]       [%]      [%/min]             [deg]       [%]      [%/min]

     350.0     97.846       0.22              450.1     93.767       0.11
     355.2     97.658       0.21              455.0     93.686       0.08
     360.1     97.484       0.20              460.0     93.634       0.04
     365.0     97.303       0.19              465.1     93.575       0.09
     370.3     97.100       0.24              470.1     93.520       0.02
     375.2     96.882       0.25              475.2     93.500       0.03
     380.0     96.658       0.22              480.1     93.483       0.02
     385.2     96.420       0.20              485.0     93.460       0.02
     390.0     96.187       0.27              490.1     93.431       0.05
     395.1     95.925       0.25              495.2     93.409       0.05
     400.1     95.680       0.26              500.2     93.398       0.02
     405.2     95.422       0.28              505.1     93.368       0.02
     410.1     95.160       0.24              510.1     93.350      -0.01
     415.1     94.912       0.27              515.2     93.335       0.00
     420.1     94.687       0.22              520.1     93.313       0.00
     425.0     94.506       0.26              525.2     93.305       0.02
     430.1     94.292       0.21              530.0     93.287       0.03
     435.1     94.118       0.19              535.1     93.261      -0.02
     440.1     93.970       0.16              540.1     93.230       0.02
     445.1     93.852       0.13              545.0     93.224       0.04
                                               550.1     93.190       0.02
```

Sample: 90% MONTMORILLONITE
Size: 26.56 mg
Rate: 15C/MIN
Program: General Analysis V1.0

TGA

Date: 12-Jul-85 Time: 10:41:40
File: PEL-RESID.42 T.LYNCH.03
Operator: JT
Plotted: 18-Jul-85 15:33:26



DuPont 1090

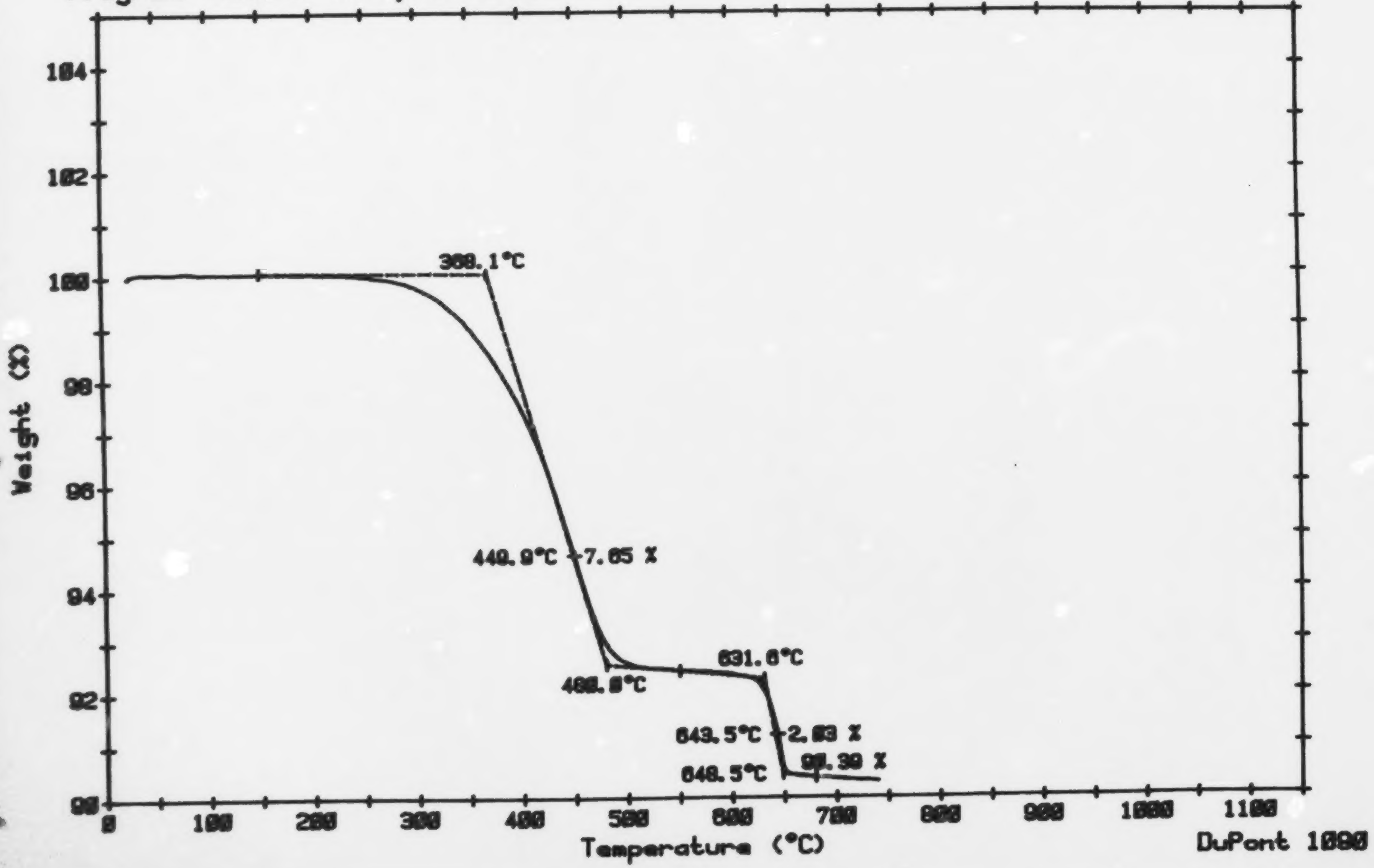
```
Sample: 90% MONTMORILLONITE                               Date: 12-Jul-85    Time: 10:41:40
Size: 26.56 mg                                            File: PEL-RESID.42 T.LYNCH.03
Rate: 15C/MIN                                             Operator: JT
Program: General Analysis V1.0                            Plotted: 18-Jul-85    15:43:48

      Temp       Sig-A       Deriv-A                     Temp       Sig-A       Deriv-A
      [deg]       [%]        [%/min]                     [deg]       [%]        [%/min]

      150.5     100.030        0.00                      250.8      99.944        0.08
      155.0     100.041       -0.02                      255.2      99.921        0.07
      160.5     100.023        0.02                      260.5      99.902        0.10
      165.8     100.030        0.04                      265.9      99.879        0.08
      170.3     100.026        0.02                      270.3      99.868        0.04
      175.6     100.015        0.00                      275.5      99.857        0.08
      180.1     100.019        0.02                      280.8      99.812        0.11
      185.4     100.004       -0.01                      285.2      99.785        0.12
      190.9     100.011        0.02                      290.4      99.740        0.16
      195.3     100.004       -0.01                      295.6      99.680        0.19
      200.6     100.000        0.00                      300.0      99.642        0.16
      205.1     100.008        0.03                      305.2      99.582        0.20
      210.5     100.004        0.05                      310.4      99.522        0.24
      215.0      99.985        0.01                      315.6      99.446        0.24
      220.4      99.985        0.01                      320.8      99.375        0.28
      225.7      99.974        0.02                      325.2      99.296        0.30
      230.2      99.977        0.02                      330.4      99.202        0.35
      235.5      99.970        0.03                      335.6      99.085        0.36
      240.0      99.966        0.02                      340.7      98.981        0.42
      245.4      99.955        0.02                      345.0      98.862        0.40
                                                         350.1      98.742        0.42
```

```
Sample: 90% MONTMORILLONITE                    Date: 12-Jul-85    Time: 10:41:40
Size: 26.56 mg                                 File: PEL-RESID.42 T.LYNCH.03
Rate: 15C/MIN                                  Operator: JT
Program: General Analysis V1.0                 Plotted: 18-Jul-85    15:51:38

     Temp      Sig-A      Deriv-A                    Temp      Sig-A      Deriv-A
     [deg]      [%]       [%/min]                    [deg]      [%]       [%/min]

     350.1     98.742       0.42                     450.2     94.272       0.98
     355.2     98.614       0.45                     455.1     93.978       0.85
     360.3     98.486       0.46                     460.7     93.715       0.74
     365.4     98.336       0.49                     465.5     93.485       0.68
     370.5     98.177       0.54                     470.2     93.320       0.54
     375.5     98.023       0.55                     475.8     93.158       0.38
     380.6     97.854       0.53                     480.5     93.058       0.30
     385.7     97.684       0.64                     485.2     92.988       0.21
     390.7     97.500       0.61                     490.7     92.921       0.14
     395.7     97.311       0.65                     495.4     92.887       0.11
     400.7     97.106       0.73                     500.7     92.849       0.06
     405.6     96.882       0.76                     505.3     92.826       0.10
     410.5     96.637       0.83                     510.7     92.808       0.08
     415.4     96.374       0.84                     515.2     92.785       0.04
     420.3     96.114       0.89                     520.4     92.774       0.05
     425.2     95.831       0.97                     525.7     92.762       0.01
     430.1     95.549       1.02                     530.3     92.744       0.06
     435.0     95.244       1.02                     535.7     92.728       0.05
     440.6     94.871       1.06                     540.2     92.710       0.01
     445.4     94.570       1.01                     545.6     92.706       0.00
                                                     550.2     92.691       0.01
```

Sample: 90% MONTMORILLONITE
Size: 27.39 mg
Rate: 20C/MIN
Program: General Analysis V1.0

TGA

Date: 11-Jul-85 Time: 10:38:53
File: PEL-RESID.43 T.LYNCH.03
Operator: JT
Plotted: 18-Jul-85 15:58:23



```
Sample: 90% MONTMORILLONITE                          Date: 11-Jul-85   Time: 10:38:53
Size: 27.39 mg                                       File: PEL-RESID.43 T.LYNCH.03
Rate: 20C/MIN                                        Operator: JT
Program: General Analysis V1.0                       Plotted: 18-Jul-85   16:08:48

      Temp       Sig-A       Deriv-A                   Temp       Sig-A      Deriv-A
      [deg]       [%]       [%/min]                    [deg]       [%]       [%/min]

      150.1     100.058       -0.01                    250.0     99.968        0.03
      155.1     100.044        0.04                    256.0     99.960        0.07
      160.1     100.047        0.01                    260.8     99.949        0.07
      166.2     100.036        0.01                    265.5     99.927        0.06
      171.1     100.040       -0.01                    270.3     99.923        0.04
      176.0     100.040        0.02                    275.0     99.916        0.09
      180.9     100.036        0.01                    280.9     99.887        0.11
      185.9     100.033        0.02                    285.6     99.865        0.13
      190.8     100.033       -0.01                    290.4     99.828        0.16
      195.6     100.033        0.01                    295.1     99.799        0.18
      200.4     100.026        0.03                    301.0     99.755        0.22
      205.2     100.022        0.02                    305.7     99.708        0.25
      210.1     100.018        0.00                    310.4     99.653        0.25
      216.1     100.026        0.00                    315.1     99.606        0.28
      220.9     100.018        0.03                    320.9     99.536        0.33
      225.6     100.015        0.01                    325.6     99.463        0.39
      230.7     100.007        0.04                    330.3     99.379        0.41
      235.5     100.000        0.03                    336.1     99.285        0.42
      240.4      99.996        0.05                    340.7     99.193        0.45
      245.2      99.982        0.09                    345.4     99.106        0.54
                                                       350.1     98.982        0.57
```

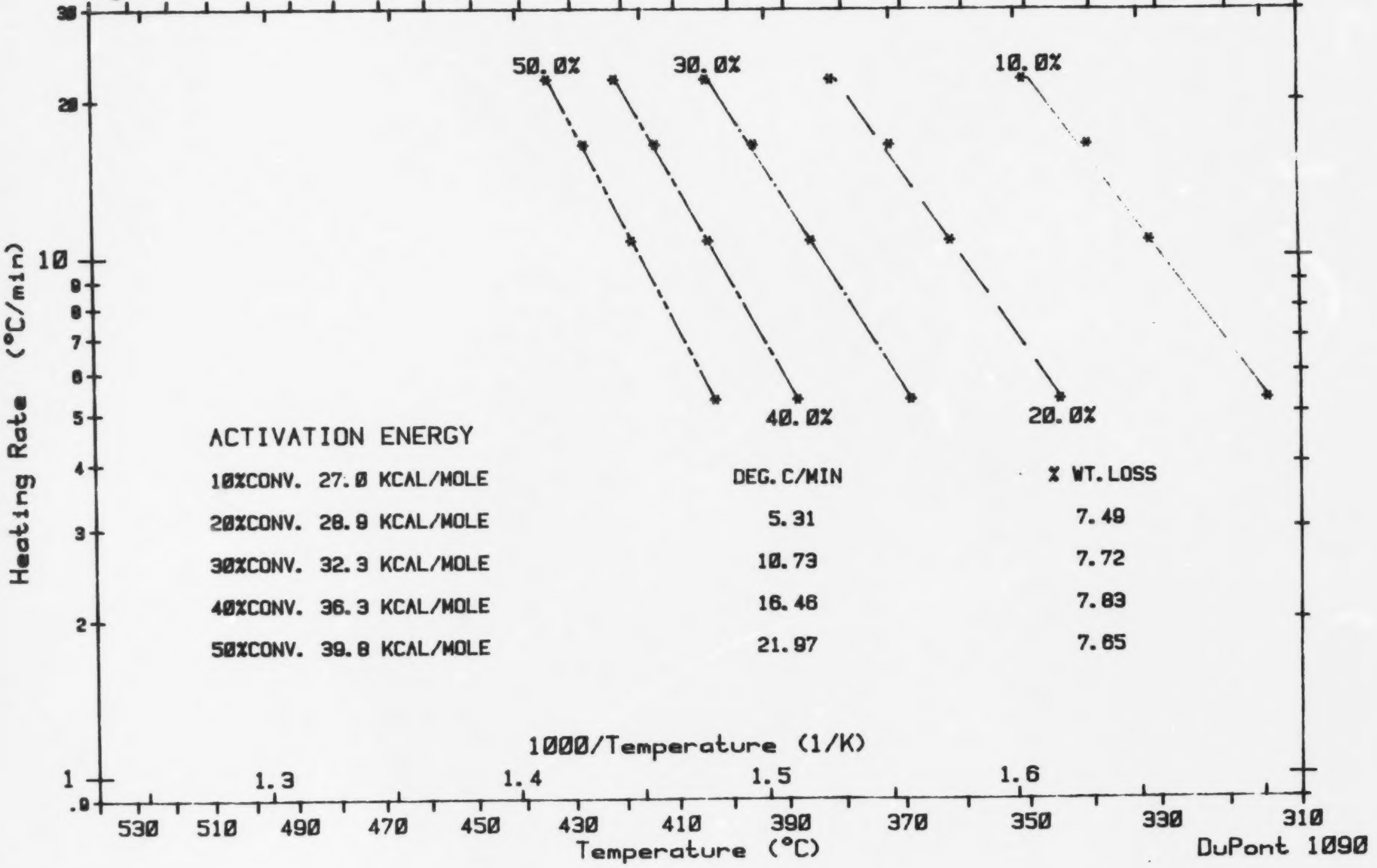
```
Sample: 90% MONTMORILLONITE                        Date: 11-Jul-85    Time: 10:38:53
Size: 27.39 mg                                     File: PEL-RESID.43 T.LYNCH.03
Rate: 20C/MIN                                      Operator: JT
Program: General Analysis V1.0                     Plotted: 18-Jul-85    16:17:10

     Temp       Sig-A      Deriv-A                  Temp       Sig-A      Deriv-A
     [deg]       [%]       [%/min]                  [deg]       [%]       [%/min]

     350.1     98.982       0.57                    451.0     94.539       1.52
     355.8     98.838       0.61                    455.3     94.232       1.48
     360.4     98.728       0.58                    460.7     93.878       1.40
     365.0     98.602       0.64                    465.0     93.608       1.25
     370.7     98.438       0.68                    470.3     93.320       1.00
     375.2     98.295       0.73                    475.6     93.075       0.87
     380.9     98.113       0.75                    480.9     92.883       0.63
     385.5     97.963       0.78                    485.2     92.783       0.48
     391.1     97.755       0.82                    490.4     92.677       0.37
     395.6     97.594       0.84                    495.7     92.612       0.22
     400.1     97.415       0.93                    501.0     92.560       0.19
     405.8     97.171       0.97                    505.1     92.528       0.14
     410.2     96.901       1.02                    510.3     92.498       0.07
     415.8     96.707       1.11                    515.6     92.487       0.07
     420.2     96.477       1.19                    520.7     92.469       0.05
     425.8     96.167       1.30                    525.9     92.462       0.00
     430.2     95.807       1.38                    530.0     92.462       0.02
     435.6     95.501       1.42                    535.2     92.455       0.07
     440.0     95.209       1.45                    540.3     92.444       0.06
     445.5     94.915       1.45                    545.4     92.425       0.06
                                                    550.5     92.411       0.03
```

Sample: 10%ILLITE 80%SAND
FILE PEL-RESID.06

Program: TGA Kinetics V1.0

Date: 20-Aug-85 Time: 16:32:44

Operator: JT
Plotted: 21-Aug-85 15:16:40



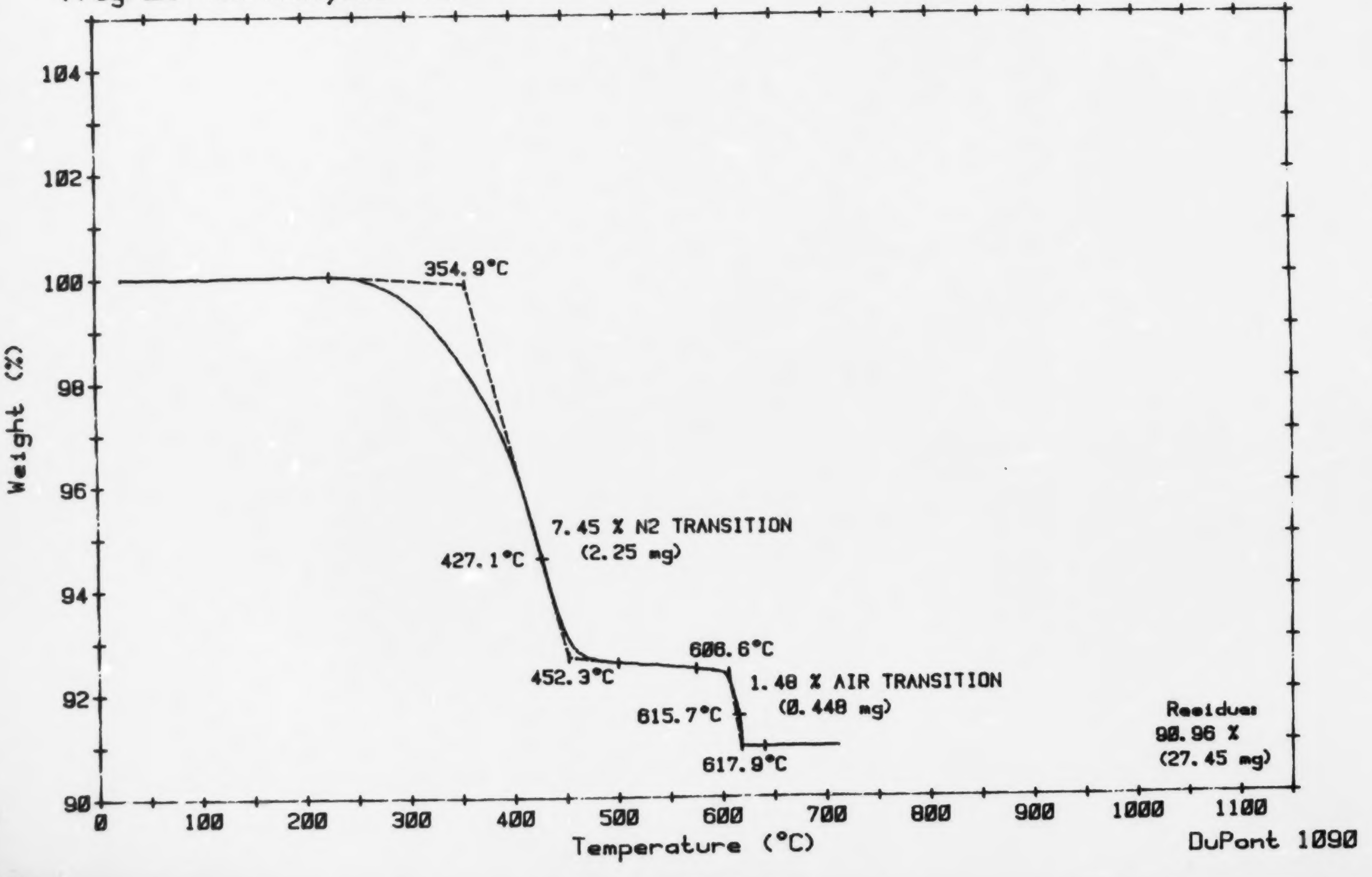
ACTIVATION ENERGY
10%CONV. 27.0 KCAL/MOLE
20%CONV. 28.9 KCAL/MOLE
30%CONV. 32.3 KCAL/MOLE
40%CONV. 36.3 KCAL/MOLE
50%CONV. 39.8 KCAL/MOLE

DEG.C/MIN	% WT.LOSS
5.31	7.49
10.73	7.72
16.46	7.83
21.97	7.65

Sample: 10%ILLITE 80%SAND
Size: 30.18 mg
Rate: 05C/MIN
Program: TGA Analysis V2.0

TGA

Date: 21-Aug-85 Time: 10:40:46
File: PEL-RESID.60 T.LYNCH.05
Operator: JT
Plotted: 9-Sep-85 13:23:31



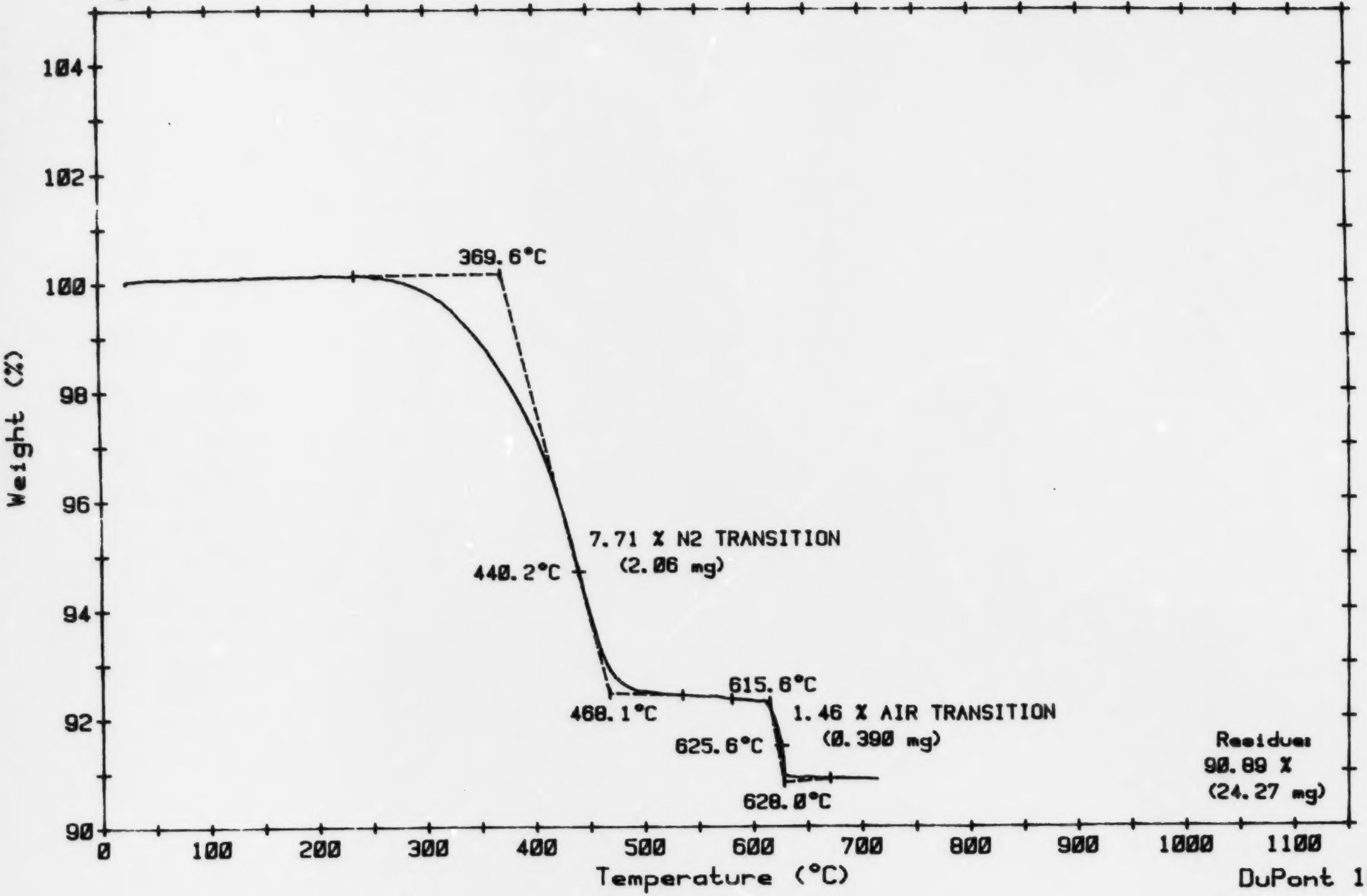
```
Sample: 10%ILLITE 80%SAND                              Date: 21-Aug-85   Time: 10:40:46
Size: 30.18 mg                                         File: PEL-RESID.60 T.LYNCH.05
Rate: 05C/MIN                                          Operator: JT
Program: General Analysis V1.0                         Plotted: 21-Aug-85   15:52:11

      Temp      Sig-A      Deriv-A                          Temp      Sig-A      Deriv-A
      [deg]      [%]       [%/min]                          [deg]      [%]       [%/min]

      200.0    100.020       0.02                           300.1     99.493       0.11
      205.0    100.013      -0.01                           305.1     99.413       0.11
      210.1    100.023      -0.02                           310.1     99.331       0.11
      215.2    100.023       0.02                           315.0     99.231       0.13
      220.0    100.027       0.01                           320.2     99.102       0.12
      225.2    100.027      -0.01                           325.1     99.003       0.14
      230.0    100.007      -0.02                           330.2     98.870       0.16
      235.0    100.010      -0.02                           335.1     98.754       0.13
      240.1    100.003      -0.01                           340.1     98.615       0.15
      245.2     99.997       0.02                           345.2     98.479       0.14
      250.2     99.983       0.00                           350.2     98.320       0.18
      255.1     99.957       0.02                           355.0     98.181       0.14
      260.0     99.920       0.05                           360.1     98.022       0.18
      265.0     99.897       0.04                           365.0     97.863       0.19
      270.0     99.854       0.05                           370.2     97.690       0.16
      275.2     99.801       0.04                           375.1     97.508       0.20
      280.2     99.751       0.07                           380.0     97.326       0.23
      285.2     99.698       0.07                           385.1     97.107       0.23
      290.1     99.639       0.09                           390.0     96.902       0.24
      295.1     99.569       0.08                           395.1     96.647       0.26
                                                            400.2     96.385       0.32
```

```
Sample: 10%ILLITE 80%SAND                              Date: 21-Aug-85    Time: 10:40:46
Size: 30.18 mg                                         File: PEL-RESID.60 T.LYNCH.05
Rate: 05C/MIN                                          Operator: JT
Program: General Analysis V1.0                         Plotted: 21-Aug-85    16:01:06

      Temp       Sig-A       Deriv-A                        Temp       Sig-A       Deriv-A
      [deg]       [%]        [%/min]                        [deg]       [%]        [%/min]

      400.2      96.385       0.32                          500.1      92.577      -0.01
      405.2      96.100       0.30                          505.1      92.581      -0.01
      410.2      95.772       0.36                          510.2      92.561       0.03
      415.0      95.440       0.39                          515.2      92.561       0.01
      420.1      95.086       0.38                          520.0      92.551       0.02
      425.2      94.721       0.39                          525.0      92.541       0.04
      430.0      94.354       0.40                          530.2      92.528       0.04
      435.0      93.992       0.36                          535.1      92.528      -0.01
      440.2      93.634       0.32                          540.0      92.514      -0.01
      445.0      93.356       0.28                          545.1      92.511       0.02
      450.2      93.114       0.22                          550.0      92.491       0.01
      455.0      92.942       0.17                          555.1      92.478       0.01
      460.2      92.809       0.14                          560.1      92.481       0.02
      465.1      92.733       0.06                          565.1      92.475       0.01
      470.2      92.690       0.01                          570.0      92.461       0.02
      475.1      92.660       0.05                          575.0      92.448       0.02
      480.1      92.630       0.03                          580.2      92.435       0.02
      485.2      92.617      -0.01                          585.2      92.428       0.01
      490.1      92.604       0.02                          590.0      92.408       0.01
      495.1      92.597       0.02                          595.1      92.402       0.01
                                                            600.4      92.375       0.16
```

Sample: 10%ILLITE 80%SAND
Size: 26.70 mg
Rate: 10C/MIN
Program: TGA Analysis V2.0

TGA

Date: 20-Aug-85 Time: 12:45:23
File: PEL-RESID.62 T.LYNCH.05
Operator: JT
Plotted: 9-Sep-85 13:14:14



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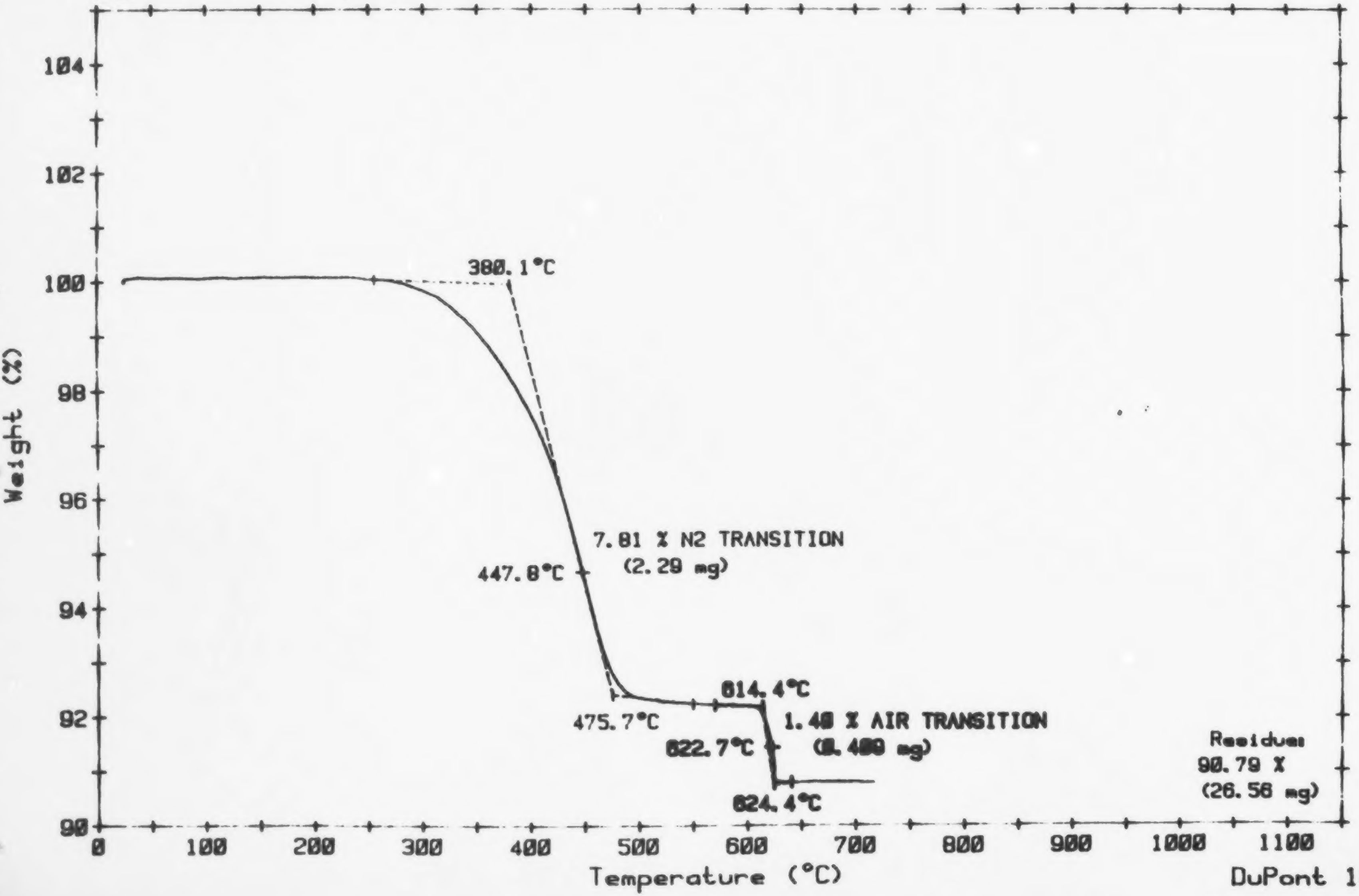
```
Sample: 10%ILLITE 80%SAND                         Date: 20-Aug-85    Time: 12:45:23
Size: 26.70 mg                                    File: PEL-RESID.62 T.LYNCH.05
Rate: 10C/MIN                                     Operator: JT
Program: General Analysis V1.0                    Plotted: 22-Aug-85   10:18:22

    Temp      Sig-A     Deriv-A                 Temp      Sig-A     Deriv-A
   [deg]       [%]     [%/min]                 [deg]       [%]     [%/min]

   200.1    100.131     -0.01                  300.1     99.839      0.11
   205.4    100.135     -0.01                  305.2     99.783      0.16
   210.0    100.135      0.03                  310.3     99.715      0.14
   215.3    100.139      0.00                  315.4     99.659      0.17
   220.0    100.139      0.00                  320.4     99.566      0.19
   225.2    100.135      0.00                  325.4     99.472      0.24
   230.4    100.127      0.00                  330.4     99.371      0.24
   235.1    100.142     -0.02                  335.4     99.262      0.23
   240.2    100.131      0.01                  340.4     99.150      0.26
   245.4    100.131      0.06                  345.2     99.038      0.26
   250.0    100.127      0.02                  350.1     98.914      0.29
   255.2    100.112      0.00                  355.1     98.794      0.29
   260.4    100.101      0.03                  360.1     98.641      0.34
   265.5    100.075      0.05                  365.0     98.502      0.31
   270.1    100.064      0.05                  370.5     98.326      0.33
   275.3    100.037      0.04                  375.4     98.173      0.34
   280.3    100.011      0.13                  380.3     98.008      0.38
   285.4     99.970      0.09                  385.2     97.832      0.40
   290.5     99.925      0.09                  390.0     97.645      0.43
   295.0     99.888      0.09                  395.4     97.424      0.46
                                               400.3     97.214      0.48
```

```
Sample: 10%ILLITE 80%SAND                                  Date: 20-Aug-85    Time: 12:45:23
Size: 26.70 mg                                             File: PEL-RESID.62 T.LYNCH.05
Rate: 10C/MIN                                              Operator: JT
Program: General Analysis V1.0                             Plotted: 22-Aug-85    10:50:42

      Temp      Sig-A      Deriv-A                           Temp      Sig-A      Deriv-A
      [deg]      [%]       [%/min]                           [deg]      [%]       [%/min]

      400.3     97.214       0.48                            500.1     92.492       0.02
      405.1     96.982       0.54                            505.2     92.484       0.04
      410.5     96.712       0.60                            510.3     92.473       0.03
      415.3     96.439       0.61                            515.4     92.451       0.08
      420.1     96.154       0.68                            520.1     92.439       0.02
      425.4     95.810       0.69                            525.3     92.436       0.02
      430.2     95.476       0.76                            530.2     92.424       0.00
      435.0     95.113       0.82                            535.1     92.436       0.01
      440.2     94.697       0.85                            540.1     92.424       0.04
      445.4     94.289       0.84                            545.1     92.432       0.00
      450.1     93.930       0.73                            550.0     92.409       0.02
      455.3     93.589       0.66                            555.0     92.402       0.00
      460.4     93.256       0.57                            560.0     92.406      -0.06
      465.1     93.024       0.45                            565.4     92.402      -0.01
      470.2     92.840       0.30                            570.4     92.387       0.08
      475.4     92.720       0.19                            575.4     92.372       0.04
      480.2     92.630       0.15                            580.4     92.357       0.02
      485.4     92.578       0.12                            585.3     92.338       0.05
      490.2     92.533       0.06                            590.2     92.331       0.02
      495.5     92.499       0.03                            595.1     92.323       0.02
                                                             600.0     92.316       0.03
```

Sample: 10%ILLITE 80%SAND
Size: 29.26 mg
Rate: 15C/MIN
Program: TGA Analysis V2.0

TGA

Date: 21-Aug-85 Time: 13:48:37
File: PEL-RESID.61 T.LYNCH.05
Operator: JT
Plotted: 9-Sep-85 12:03:09



DuPont 1090

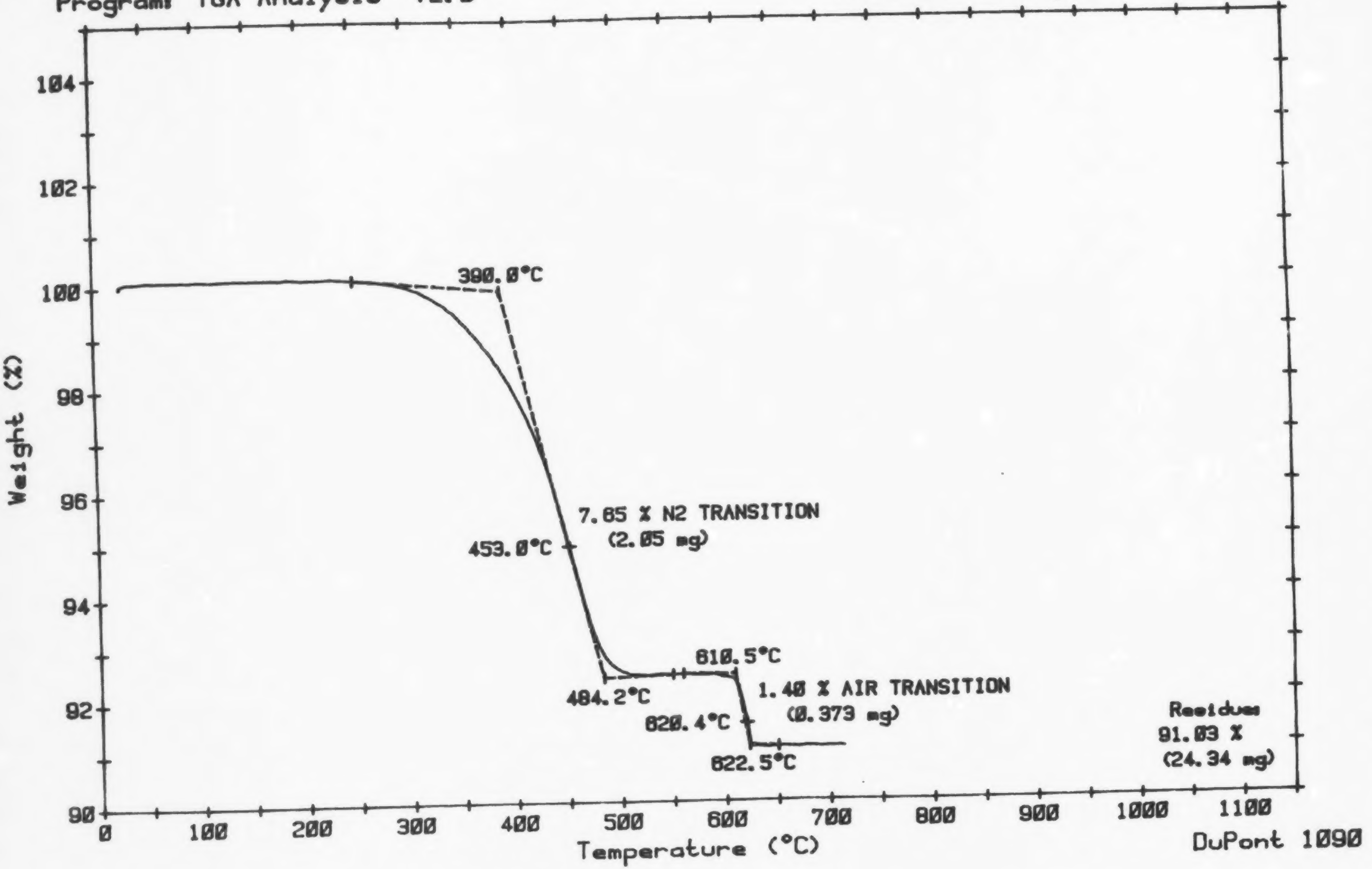
```
Sample: 10%ILLITE 80%SAND                          Date: 21-Aug-85    Time: 13:48:37
Size: 29.26 mg                                     File: PEL-RESID.61 T.LYNCH.05
Rate: 15C/MIN                                      Operator: JT
Program: General Analysis V1.0                     Plotted: 22-Aug-85    11:10:30

      Temp       Sig-A      Deriv-A                     Temp      Sig-A     Deriv-A
      [deg]       [%]       [%/min]                     [deg]      [%]      [%/min]

      200.2     100.075       0.05                      300.8     99.829      0.13
      205.6     100.079      -0.03                      305.1     99.785      0.17
      210.1     100.079       0.00                      310.3     99.744      0.14
      215.5     100.082      -0.01                      315.5     99.692      0.26
      220.0     100.079       0.00                      320.6     99.607      0.24
      225.3     100.082      -0.01                      325.7     99.532      0.30
      230.6     100.089      -0.01                      330.8     99.436      0.31
      235.1     100.082       0.04                      335.0     99.378      0.27
      240.4     100.065       0.05                      340.1     99.279      0.35
      245.7     100.048       0.03                      345.1     99.176      0.35
      250.1     100.051       0.03                      350.1     99.057      0.40
      255.4     100.034       0.00                      355.1     98.934      0.42
      260.8     100.051       0.02                      360.2     98.814      0.43
      265.2     100.034       0.04                      365.1     98.674      0.46
      270.4     100.007       0.06                      370.2     98.534      0.49
      275.7      99.993       0.08                      375.3     98.383      0.51
      280.1      99.969       0.07                      380.3     98.219      0.54
      285.3      99.949       0.14                      385.4     98.086      0.55
      290.5      99.904       0.12                      390.4     97.888      0.60
      295.6      99.867       0.12                      395.4     97.714      0.60
                                                        400.4     97.519      0.64
```

```
Sample: 10%ILLITE 80%SAND                          Date: 21-Aug-85    Time: 13:48:37
Size: 29.26 mg                                     File: PEL-RESID.61 T.LYNCH.05
Rate: 15C/MIN                                      Operator: JT
Program: General Analysis V1.0                     Plotted: 22-Aug-85    11:52:46

     Temp       Sig-A      Deriv-A                     Temp       Sig-A      Deriv-A
     [deg]       [%]       [%/min]                     [deg]       [%]       [%/min]

     400.4     97.519        0.64                      500.4     92.327        0.08
     405.4     97.310        0.74                      505.1     92.314        0.04
     410.4     97.085        0.75                      510.5     92.303        0.04
     415.3     96.842        0.86                      515.1     92.286        0.05
     420.2     96.576        0.94                      520.5     92.258        0.05
     425.2     96.282        1.00                      525.1     92.249        0.00
     430.0     95.971        1.12                      530.4     92.245        0.05
     435.7     95.564        1.15                      535.0     92.242        0.01
     440.6     95.208        1.23                      540.3     92.239       -0.01
     445.4     94.836        1.31                      545.6     92.242        0.05
     450.3     94.443        1.28                      550.1     92.221        0.01
     455.1     94.050        1.28                      555.3     92.211        0.06
     460.8     93.616        1.19                      560.4     92.204        0.02
     465.6     93.288        1.02                      565.5     92.201        0.01
     470.4     93.011        0.84                      570.3     92.191        0.01
     475.2     92.782        0.60                      575.7     92.198       -0.01
     480.8     92.601        0.46                      580.0     92.204        0.01
     485.5     92.488        0.34                      585.0     92.201       -0.02
     490.2     92.403        0.18                      590.1     92.194        0.03
     495.7     92.362        0.13                      595.2     92.180        0.01
                                                       600.2     92.187        0.05
```

Sample: 10%ILLITE 80%SAND
Size: 26.74 mg
Rate: 20C/MIN
Program: TGA Analysis V2.0

TGA

Date: 20-Aug-85 Time: 16:32:44
File: PEL-RESID.64 T.LYNCH.05
Operator: JT
Plotted: 9-Sep-85 13:36:11



DuPont 1090

```
Sample: 10%ILLITE 80%SAND                                  Date: 20-Aug-85    Time: 16:32:44
Size: 26.74 mg                                             File: PEL-RESID.64 T.LYNCH.05
Rate: 20C/MIN                                              Operator: JT
Program: General Analysis V1.0                             Plotted: 22-Aug-85    15:49:38

        Temp       Sig-A      Deriv-A                      Temp       Sig-A      Deriv-A
       [deg]        [%]       [%/min]                      [deg]       [%]       [%/min]

       200.9     100.105       0.01                        301.1     99.948       0.13
       205.6     100.112      -0.01                        305.8     99.910       0.18
       210.3     100.112       0.00                        310.4     99.880       0.18
       215.0     100.112       0.00                        315.0     99.835       0.25
       220.9     100.116      -0.01                        320.8     99.772       0.25
       225.6     100.120      -0.02                        325.4     99.719       0.28
       230.4     100.120       0.00                        331.1     99.648       0.28
       235.1     100.120       0.05                        335.7     99.589       0.33
       241.1     100.105       0.04                        340.3     99.518       0.36
       245.8     100.093       0.04                        345.9     99.424       0.43
       250.5     100.090       0.02                        350.5     99.323       0.48
       255.2     100.090       0.05                        355.0     99.230       0.48
       261.1     100.071       0.01                        360.7     99.110       0.54
       265.8     100.075       0.04                        365.3     98.984       0.58
       270.5     100.056       0.06                        370.9     98.858       0.58
       275.3     100.052       0.06                        375.4     98.736       0.58
       281.1     100.034       0.06                        381.0     98.590       0.68
       285.8     100.019       0.08                        385.5     98.444       0.69
       290.5      99.986       0.12                        391.0     98.265       0.73
       295.2      99.974       0.12                        395.4     98.122       0.75
                                                           400.9     97.913       0.86
```

```
Sample: 10%ILLITE 80%SAND                              Date: 20-Aug-85    Time: 16:32:44
Size: 26.74 mg                                         File: PEL-RESID.64 T.LYNCH.05
Rate: 20C/MIN                                          Operator: JT
Program: General Analysis V1.0                         Plotted: 22-Aug-85   15:57:39

     Temp      Sig-A      Deriv-A                      Temp      Sig-A      Deriv-A
     [deg]      [%]       [%/min]                      [deg]      [%]       [%/min]

     400.9     97.913      0.86                        500.2     92.523      0.22
     405.3     97.752      0.88                        505.3     92.482      0.18
     410.8     97.528      0.92                        510.5     92.449      0.07
     415.2     97.344      0.99                        515.7     92.437      0.03
     420.6     97.079      1.07                        520.8     92.430      0.03
     426.1     96.787      1.20                        526.0     92.437      0.02
     430.4     96.544      1.28                        530.1     92.430     -0.01
     435.8     96.211      1.43                        535.2     92.430     -0.03
     440.1     95.905      1.56                        540.3     92.430      0.00
     445.5     95.516      1.62                        545.5     92.434     -0.02
     450.8     95.089      1.73                        550.6     92.437     -0.02
     455.1     94.741      1.71                        555.7     92.437     -0.01
     460.4     94.322      1.68                        560.8     92.449      0.00
     465.7     93.904      1.62                        565.8     92.434      0.06
     471.0     93.526      1.41                        570.7     92.426     -0.02
     475.1     93.257      1.21                        575.8     92.426      0.03
     480.4     92.991      0.94                        580.9     92.422     -0.01
     485.5     92.785      0.86                        585.9     92.434     -0.03
     490.7     92.662      0.43                        590.8     92.430      0.04
     495.9     92.576      0.29                        595.6     92.400      0.14
                                                       600.0     92.374      0.02
```

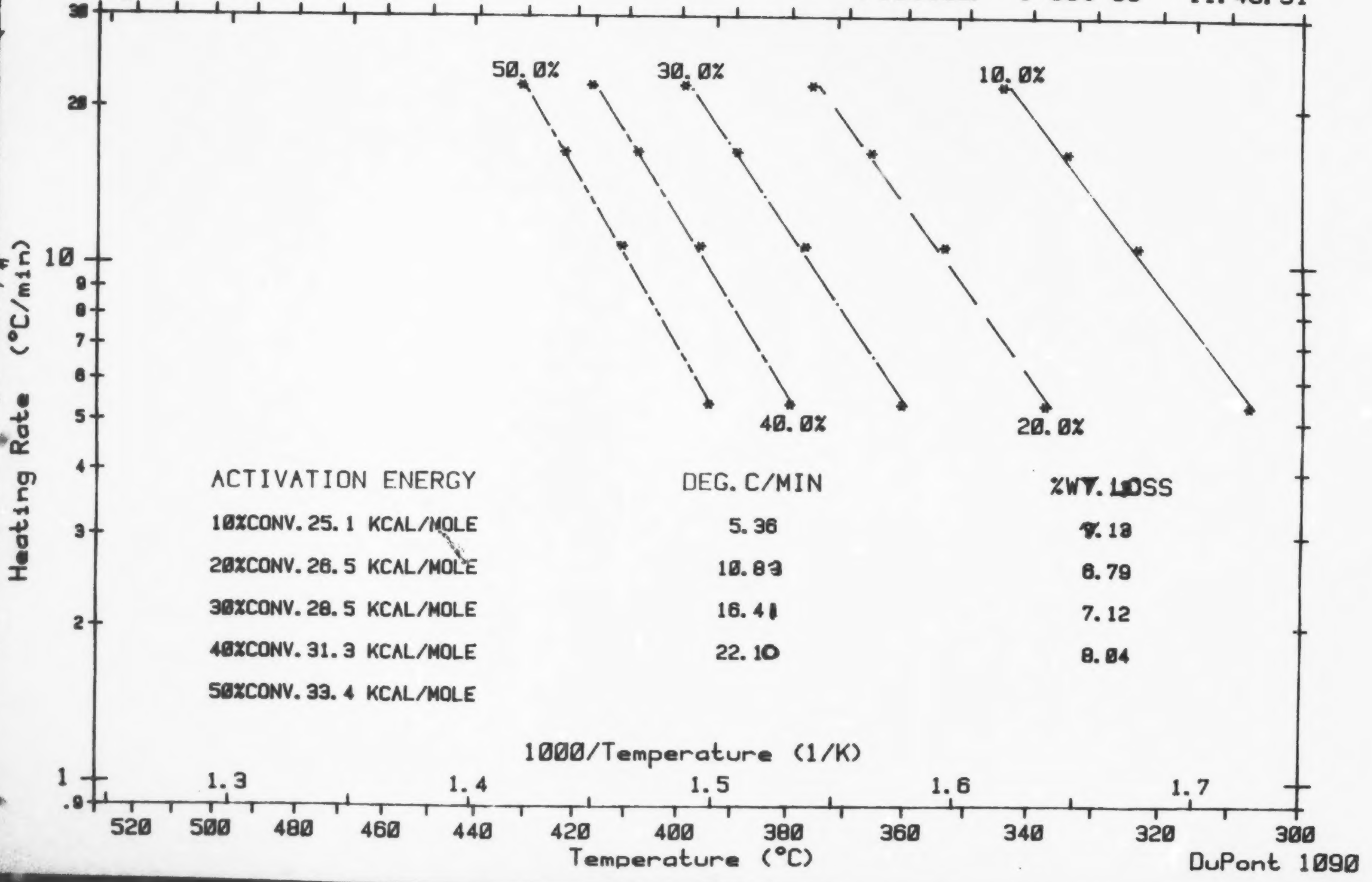
Sample: 45% ILLITE

Date: 27-Sep-85 Time: 14:56:55

Operator: JT

Program: TGA Kinetics V1.0

Plotted: 1-Oct-85 11:48:51



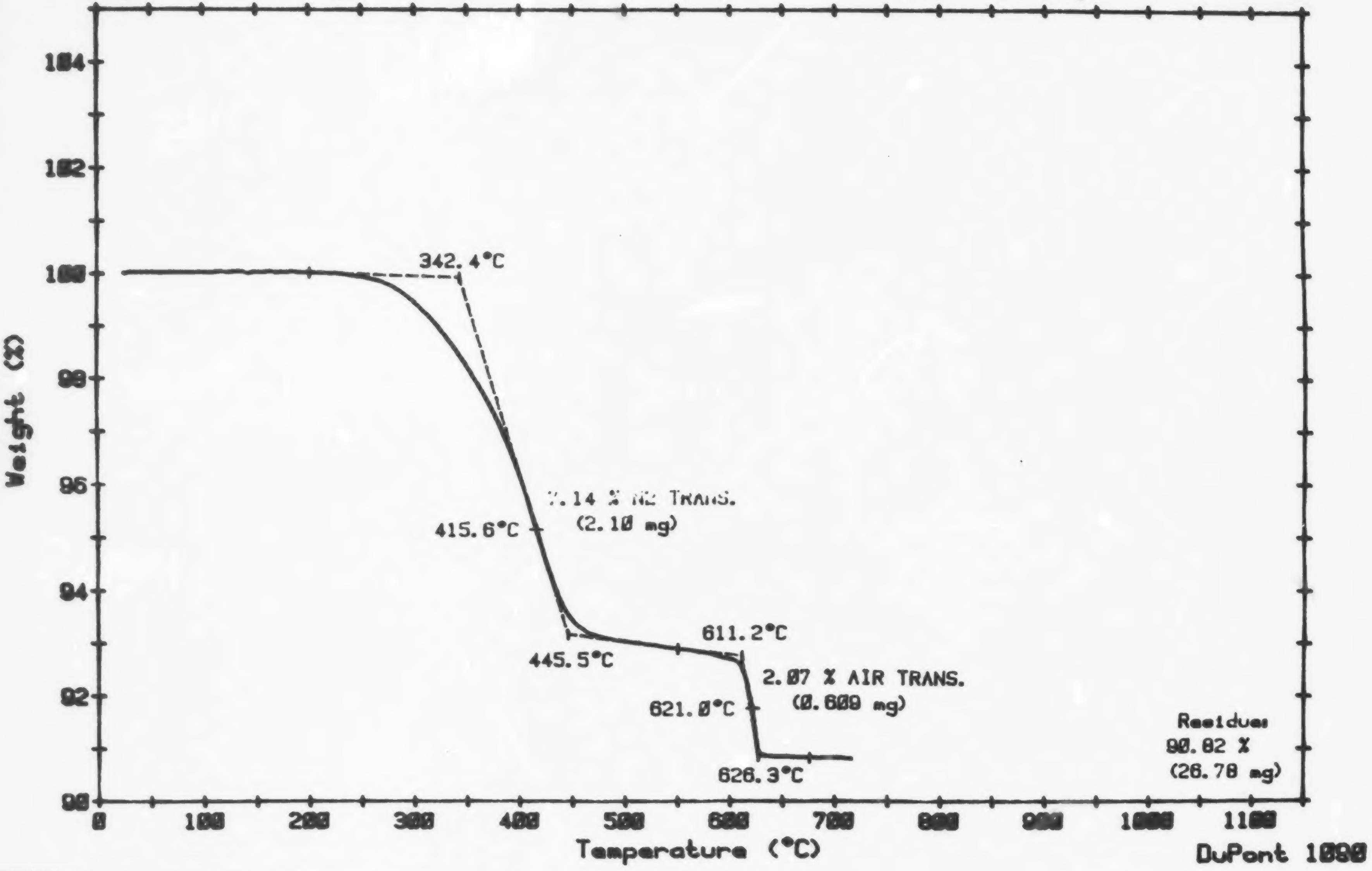
ACTIVATION ENERGY	DEG. C/MIN	%WT. LOSS
10%CONV. 25.1 KCAL/MOLE	5.36	7.18
20%CONV. 26.5 KCAL/MOLE	10.83	6.79
30%CONV. 28.5 KCAL/MOLE	16.41	7.12
40%CONV. 31.3 KCAL/MOLE	22.10	8.04
50%CONV. 33.4 KCAL/MOLE		

DuPont 1090

Sample: 45% ILLITE
Size: 29.49 mg
Rate: 05C/MIN
Program: TGA Analysis V2.0

TGA

Date: 26-Sep-85 Time: 16:06:58
File: PEL/RESID.10 T.LYNCH.00
Operator: JT
Plotted: 27-Sep-85 15:59:30

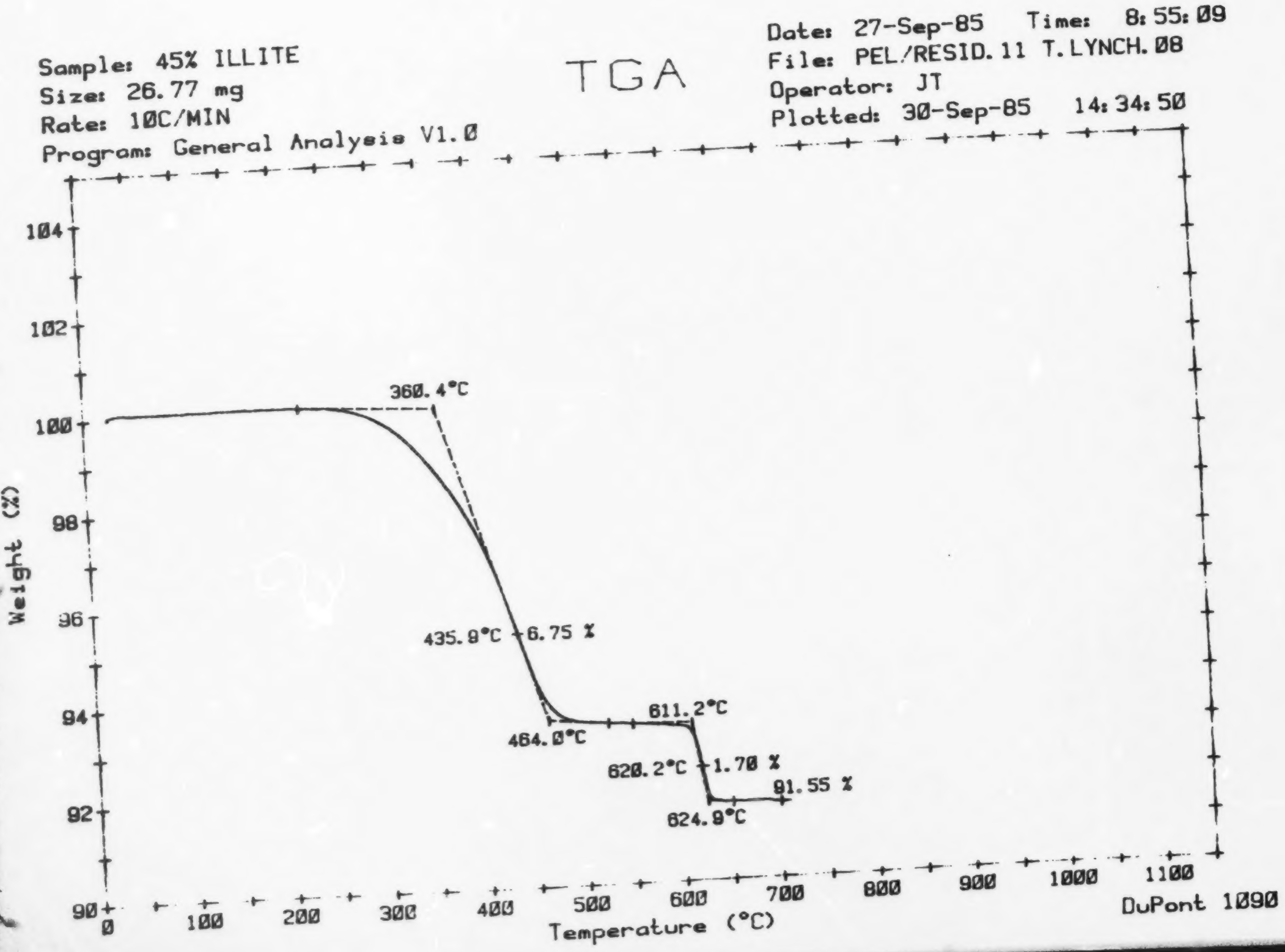


```
Sample: 45% ILLITE                                    Date: 26-Sep-85   Time: 16:06:58
Size: 29.49 mg                                        File: PEL/RESID.10 T.LYNCH.08
Rate: 05C/MIN                                         Operator: JT
Program: General Analysis V1.0                        Plotted: 30-Sep-85   13:31:13

     Temp       Sig-A       Deriv-A                        Temp       Sig-A       Deriv-A
     [deg]       [%]        [%/min]                        [deg]       [%]        [%/min]

     150.0     100.044        0.00                         250.2      99.959        0.01
     155.2     100.047        0.00                         255.1      99.942        0.02
     160.0     100.047        0.00                         260.1      99.919        0.01
     165.2     100.044        0.00                         265.1      99.888        0.01
     170.2     100.054        0.00                         270.0      99.854        0.02
     175.0     100.051        0.00                         275.1      99.810        0.03
     180.1     100.051        0.00                         280.2      99.769        0.03
     185.1     100.041        0.01                         285.0      99.708        0.03
     190.2     100.044        0.00                         290.2      99.630        0.02
     195.2     100.047        0.00                         295.1      99.559        0.05
     200.2     100.044        0.01                         300.2      99.468        0.05
     205.2     100.044        0.00                         305.1      99.373        0.06
     210.0     100.034        0.00                         310.0      99.281        0.05
     215.0     100.031        0.00                         315.2      99.186        0.05
     220.0   [illegible]   [illegible]                     320.1      99.064        0.06
     225.1     100.027        0.00                         325.2      98.932        0.08
     230.1     100.007        0.01                         330.1      98.810        0.08
     235.1     100.000        0.00                         335.2      98.688        0.07
     240.2      99.983        0.01                         340.1      98.545        0.08
     245.2      99.969        0.02                         345.2      98.389        0.09
                                                           350.0      98.253        0.09
```

Sample: 45% ILLITE
Size: 29.49 mg
Rate: 05C/MIN
Program: General Analysis V1.0

Date: 26-Sep-85 Time: 16:06:58
File: PEL/RESID.10 T.LYNCH.08
Operator: JT
Plotted: 30-Sep-85 13:44:39

Temp [deg]	Sig-A [%]	Deriv-A [%/min]
350.0	98.253	0.09
355.1	98.084	0.09
360.2	97.921	0.11
365.1	97.758	0.09
370.1	97.572	0.10
375.2	97.372	0.11
380.0	97.175	0.12
385.1	96.938	0.12
390.2	96.697	0.13
395.2	96.436	0.14
400.0	96.168	0.16
405.1	95.866	0.17
410.0	95.547	0.17
415.1	95.204	0.18
420.1	94.8[illegible]	[illegible]
425.0	94.547	0.16
430.0	94.258	0.15
435.0	93.990	0.13
440.2	93.750	0.11
445.2	93.570	0.08
450.2	93.441	0.05
455.1	93.336	0.04
460.2	93.261	0.03
465.1	93.204	0.03
470.1	93.156	0.02
475.0	93.139	0.02
480.0	93.109	0.02
485.1	93.088	0.01
490.2	93.064	0.01
495.2	93.058	-0.01
500.1	93.037	0.01
505.2	93.031	0.01
510.0	93.014	0.01
515.1	93.007	0.01
520.0	92.980	0.01
525.1	92.966	0.01
530.2	92.963	0.00
535.2	92.946	0.00
540.2	92.925	-0.01
545.2	92.919	0.01
550.1	92.908	0.00



Sample: 45% ILLITE
Size: 26.77 mg
Rate: 10C/MIN
Program: General Analysis V1.0

TGA

Date: 27-Sep-85 Time: 8:55:09
File: PEL/RESID.11 T.LYNCH.08
Operator: JT
Plotted: 30-Sep-85 14:34:50

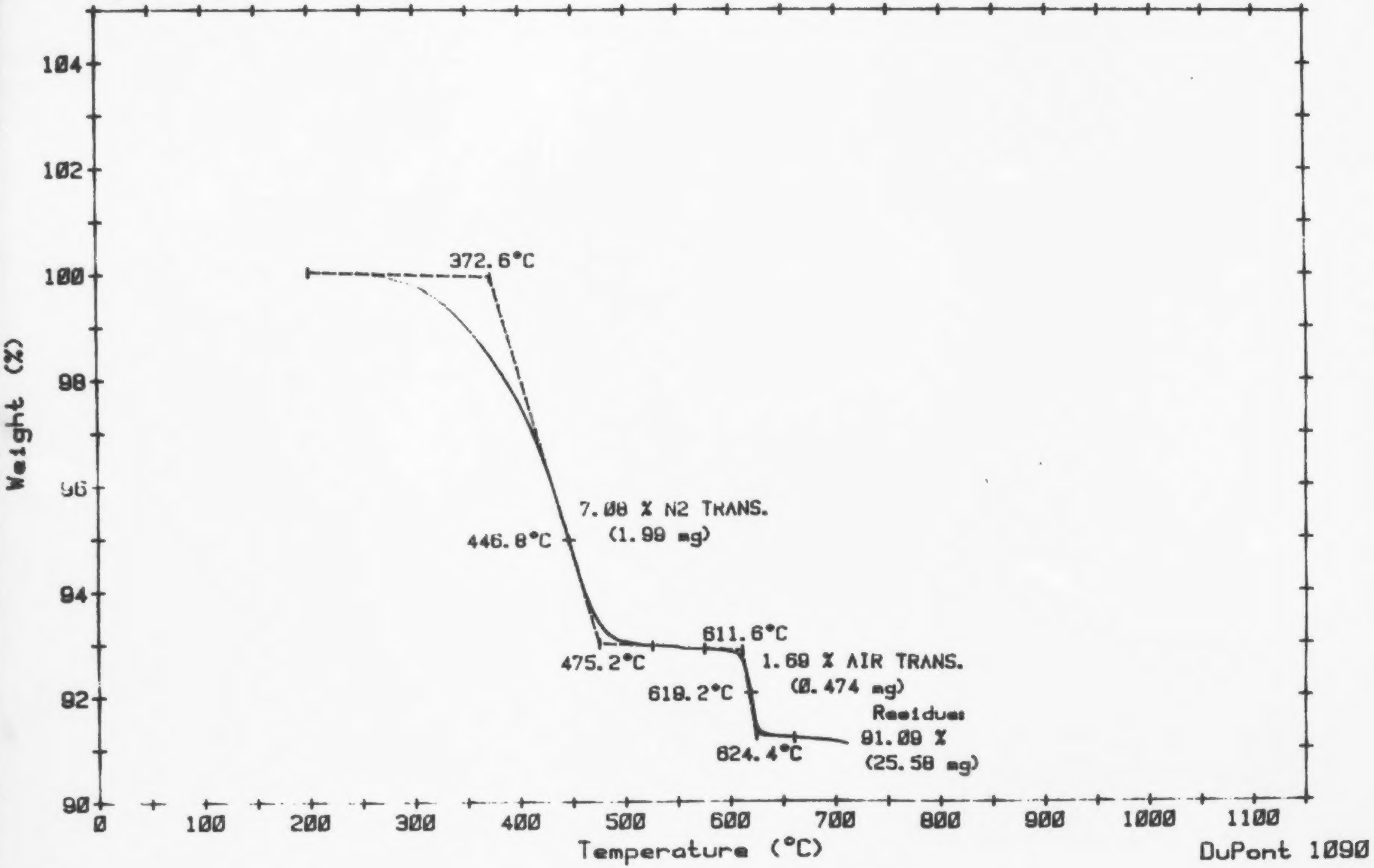
```
Sample: 45% ILLITE                                       Date: 27-Sep-85    Time:   8:55:09
Size: 26.77 mg                                           File: PEL/RESID.11 T.LYNCH.08
Rate: 10C/MIN                                            Operator: JT
Program: General Analysis V1.0                           Plotted: 30-Sep-85    15:24:01

      Temp       Sig-A      Deriv-A                    Temp       Sig-A      Deriv-A
      [deg]       [%]      [%/min]                     [deg]       [%]      [%/min]

      150.1     100.078       0.00                     250.5     100.030       0.01
      155.4     100.086       0.00                     255.0     100.019       0.02
      160.1     100.082       0.00                     260.0     100.015       0.00
      165.4     100.086       0.00                     265.1      99.985       0.02
      170.0     100.078       0.02                     270.1      99.966       0.04
      175.2     100.075      -0.01                     275.1      99.940       0.04
      180.5     100.075      -0.01                     280.1      99.910       0.04
      185.0     100.078       0.02                     285.1      99.877       0.03
      190.2     100.075      -0.01                     290.2      99.832       0.05
      195.3     100.078      -0.01                     295.2      99.787       0.05
      200.5     100.075       0.01                     300.1      99.735       0.07
      205.1     100.078       0.00                     305.1      99.668       0.08
      210.2     100.075      -0.01                     310.2      99.612       0.07
      215.4     100.075       0.00                     315.1      99.537       0.10
      220.5   [illegible]  [illegible]                 320.2      99.458       0.09
      225.1     100.063      -0.02                     325.1      99.373       0.10
      230.2     100.063       0.01                     330.0      99.275       0.10
      235.3     100.049       0.01                     335.5      99.163       0.12
      240.4     100.037      -0.01                     340.4      99.048       0.13
      245.4     100.049       0.01                     345.3      98.928       0.13
                                                       350.1      98.809       0.13
```

```
Sample: 45% ILLITE                              Date: 27-Sep-85    Time:  8:55:09
Size:  26.77 mg                                 File: PEL/RESID.11 T.LYNCH.08
Rate:  10C/MIN                                  Operator:  JT
Program: General Analysis V1.0                  Plotted: 30-Sep-85   15:36:35

     Temp       Sig-A      Deriv-A                  Temp       Sig-A      Deriv-A
     [deg]      [%]        [%/min]                  [deg]      [%]        [%/min]

     350.1      98.809      0.13                    450.0      94.349      0.28
     355.1      98.693      0.15                    455.2      94.095      0.23
     360.0      98.555      0.15                    460.5      93.882      0.20
     365.3      98.398      0.16                    465.2      93.740      0.14
     370.2      98.256      0.13                    470.3      93.621      0.10
     375.0      98.118      0.18                    475.5      93.520      0.06
     380.4      97.942      0.17                    480.2      93.464      0.05
     385.2      97.770      0.21                    485.3      93.426      0.03
     390.5      97.580      0.20                    490.5      93.400      0.03
     395.3      97.400      0.21                    495.1      93.378      0.02
     400.1      97.199      0.23                    500.2      93.363      0.01
     405.4      96.963      0.25                    505.4      93.344      0.03
     410.1      96.728      0.27                    510.0      93.341      0.01
     415.3      96.452      0.29                    515.1      93.341      0.00
     420.1      96.165      0.30                    520.1      93.326      0.01
     425.4      95.869      0.33                    525.2      93.322      0.02
     430.1      95.574      0.32                    530.2      93.318      0.00
     435.4      95.249      0.33                    535.3      93.314      0.01
     440.0      94.947      0.33                    540.3      93.282      0.01
     445.3      94.618      0.31                    545.3      93.288      0.01
                                                    550.3      93.285      0.01
```

Sample: 45% ILLITE
Size: 28.08 mg
Rate: 15C/MIN
Program: TGA Analysis V2.0

TGA

Date: 27-Sep-85 Time: 13:37:22
File: PEL/RESID.12 T.LYNCH.08
Operator: JT
Plotted: 27-Sep-85 14:32:36



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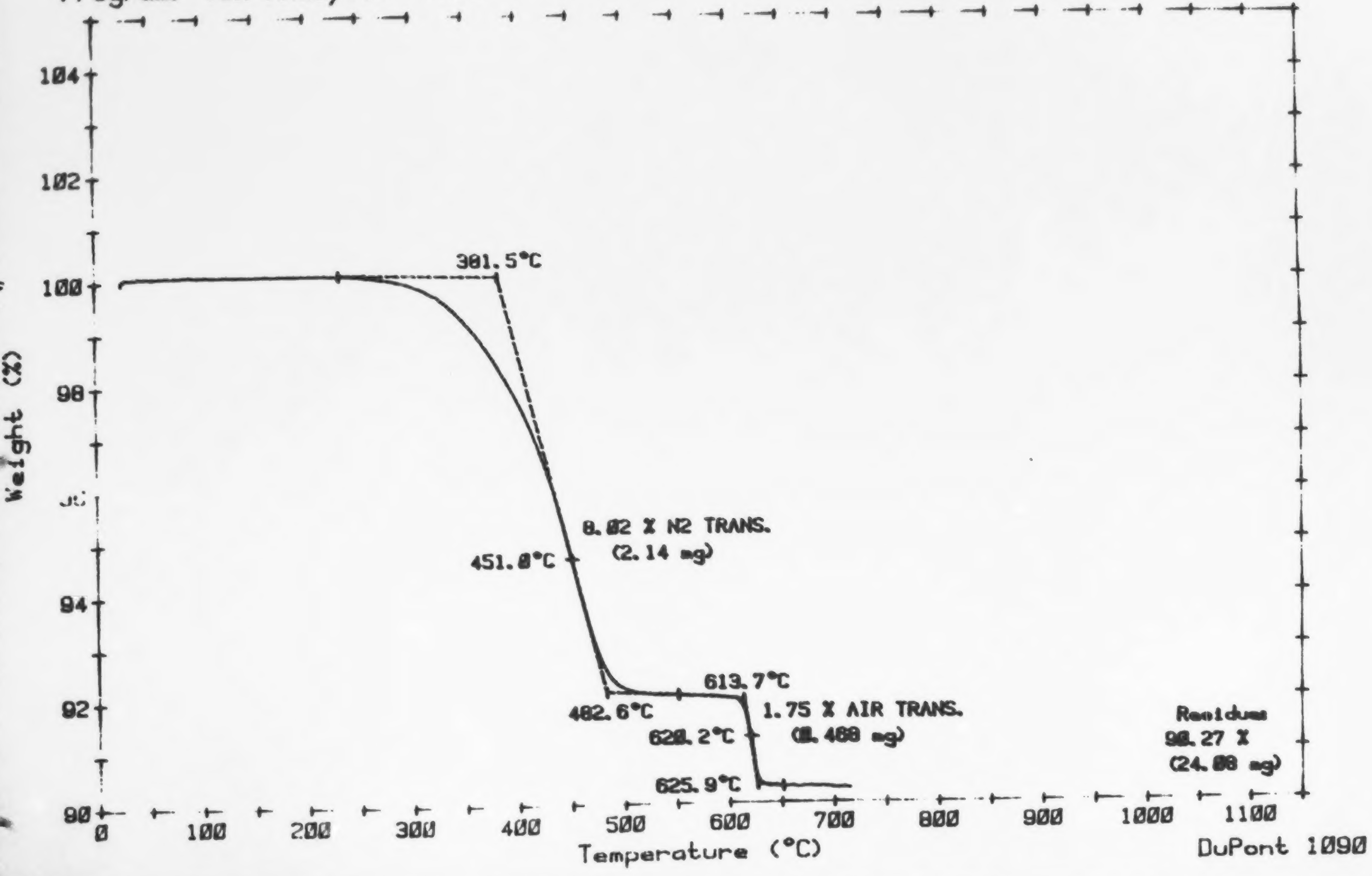
```
Sample:  45% ILLITE                                       Date:  27-Sep-85    Time:  13:37:22
Size:  28.08 mg                                           File:  PEL./RESID.12  T.LYNCH.08
Rate:  15C/MIN                                            Operator:  JT
Program:  General Analysis V1.0                           Plotted:   1-Oct-85      8:23:02

     Temp        Sig-A       Deriv-A                           Temp       Sig-A       Deriv-A
    [deg]         [%]        [%/min]                          [deg]        [%]        [%/min]

    150.0      100.050       -0.01                            250.5      100.011        0.02
    155.3      100.053        0.00                            255.6      100.007        0.00
    160.7      100.057        0.01                            260.7      100.000        0.01
    165.2      100.061        0.00                            265.8       99.989        0.03
    170.5      100.061        0.00                            270.0       99.968        0.04
    175.8      100.064        0.01                            275.0       99.950        0.04
    180.1      100.061        0.00                            280.0       99.918        0.04
    185.4      100.061        0.00                            285.0       99.893        0.05
    190.7      100.050        0.01                            290.1       99.872        0.03
    195.1      100.053        0.00                            295.2       99.843        0.05
    200.3      100.053       -0.01                            300.2       99.808        0.07
    205.5      100.043        0.02                            305.2       99.765        0.08
    210.7      100.046       -0.01                            310.1       99.697        0.11
    215.1      100.050        0.00                            315.0       99.637        0.11
    220.3      100.040        0.00                            320.0       99.573        0.11
    225.5      100.043        0.02                            325.0       99.498        0.13
    230.7      100.032        0.01                            330.1       99.420        0.14
    235.0      100.028        0.01                            335.7       99.320        0.14
    240.1      100.028       -0.01                            340.6       99.220        0.18
    245.3      100.025        0.02                            345.6       99.113        0.19
                                                              350.6       98.999        0.18
```

```
Sample: 45% ILLITE                                 Date: 27-Sep-85   Time: 13:37:22
Size: 28.08 mg                                     File: PEL/RESID.12 T.LYNCH.08
Rate: 15C/MIN                                      Operator: JT
Program: General Analysis V1.0                     Plotted:  1-Oct-85    9:44:00

     Temp       Sig-A      Deriv-A                      Temp      Sig-A      Deriv-A
    [deg]        [%]       [%/min]                     [deg]       [%]       [%/min]

    350.6      98.999       0.18                       450.8     94.680       0.54
    355.5      98.889       0.20                       455.5     94.366       0.52
    360.5      98.764       0.21                       460.2     94.078       0.45
    365.4      98.640       0.21                       465.0     93.822       0.39
    370.3      98.511       0.23                       470.6     93.585       0.33
    375.1      98.362       0.24                       475.3     93.394       0.24
    380.0      98.212       0.25                       480.0     93.263       0.19
    385.7      98.034       0.26                       485.5     93.156       0.11
    390.6      97.881       0.27                       490.2     93.102       0.09
    395.5      97.710       0.28                       495.7     93.063       0.03
    400.3      97.536       0.33                       500.3     93.038       0.03
    405.1      97.336       0.34                       505.0     93.024       0.03
    410.1      97.119       0.36                       510.4     93.010       0.02
    415.6      96.849       0.41                       515.1     92.988       0.03
    420.4      96.606       0.42                       520.3     92.978       0.01
    425.3      96.343       0.45                       525.7     92.971       0.00
    430.1      96.062       0.48                       530.3     92.974       0.01
    435.7      95.716       0.51                       535.6     92.963       0.01
    440.4      95.381       0.53                       540.2     92.956       0.01
    445.2      95.075       0.55                       545.4     92.942       0.02
                                                       550.0     92.931       0.02
```

Sample: 45% ILLITE
Size: 26.68 mg
Rate: 20C/MIN
Program: TGA Analysis V2.0

TGA

Date: 27-Sep-85 Time: 14:56:55
File: PEL/RESID.13 T.LYNCH.08
Operator: JT
Plotted: 27-Sep-85 15:41:51



DuPont 1090

```
Sample: 45% ILLITE                                     Date: 27-Sep-85    Time: 14:56:55
Size: 26.68 mg                                         File: PEL/RESID.13 T.LYNCH.08
Rate: 20C/MIN                                          Operator: JT
Program: General Analysis V1.0                         Plotted:  3-Oct-85   10:08:59

     Temp      Sig-A      Deriv-A                           Temp      Sig-A      Deriv-A
     [deg]      [%]       [%/min]                           [deg]      [%]       [%/min]

     150.3    100.094      0.00                             250.5    100.075      0.03
     156.1    100.097     -0.02                             255.1    100.056      0.08
     160.8    100.097      0.01                             260.8    100.034      0.06
     165.5    100.097      0.00                             265.4    100.026      0.05
     170.3    100.097      0.00                             270.0    100.011      0.07
     175.0    100.097      0.03                             275.7     99.993      0.05
     180.8    100.086      0.04                             280.2     99.985      0.07
     185.5    100.079      0.00                             285.9     99.951      0.10
     190.1    100.090     -0.03                             290.4     99.936      0.11
     196.0    100.097      0.01                             296.1     99.910      0.13
     200.7    100.094     -0.02                             300.7     99.880      0.17
     205.4    100.097      0.02                             305.2     99.839      0.18
     210.1    100.094      0.01                             310.8     99.794      0.22
     215.9    100.090      0.03                             315.2     99.741      0.25
     220.4    [illegible]  [illegible]                      320.8     99.689      0.23
     225.1    100.082      0.00                             325.3     99.640      0.33
     230.8    100.079      0.00                             330.9     99.550      0.36
     235.5    100.082      0.02                             335.4     99.468      0.40
     240.1    100.071      0.04                             340.8     99.370      0.43
     245.9    100.067      0.00                             345.3     99.277      0.47
                                                            350.8     99.157      0.50
```

```
Sample:  45% ILLITE                                   Date:  27-Sep-85    Time:  14:56:55
Size:  26.58 mg                                       File:  PEL/RESID.13 T.LYNCH.08
Rate:  20C/MIN                                        Operator:  JT
Program:  General Analysis V1.0                       Plotted:   3-Oct-85    10:34:16

    Temp       Sig-A       Deriv-A                    Temp       Sig-A       Deriv-A
    [deg]       [%]       [%/min]                     [deg]       [%]        [%/min]

    350.8     99.157        0.50                      451.0     94.640        1.70
    355.2     99.059        0.50                      455.2     94.288        1.70
    360.7     98.924        0.56                      460.5     93.876        1.64
    365.1     98.812        0.59                      465.8     93.482        1.49
    370.6     98.651        0.65                      470.1     93.197        1.35
    375.0     98.520        0.67                      475.4     92.883        1.16
    380.6     98.340        0.73                      480.6     92.624        0.87
    386.1     98.160        0.75                      485.9     92.444        0.63
    390.4     98.006        0.77                      490.1     92.332        0.46
    395.9     97.815        0.80                      495.4     92.242        0.29
    400.3     97.650        0.85                      500.6     92.189        0.17
    405.7     97.425        0.93                      505.8     92.148        0.14
    410.0     97.226        1.00                      511.0     92.118        0.10
    415.5     96.968        1.10                      515.2     92.107        0.05
    420.9     [illegible]   [illegible]               520.4     92.092        0.07
    425.2     96.439        1.25                      525.5     92.084        0.07
    430.5     96.113        1.35                      530.6     92.066        0.03
    436.0     95.701        1.47                      535.7     92.062        0.02
    440.3     95.457        1.57                      540.7     92.058        0.00
    445.6     95.053        1.67                      545.8     92.054        0.01
                                                      551.0     92.054        0.03
```

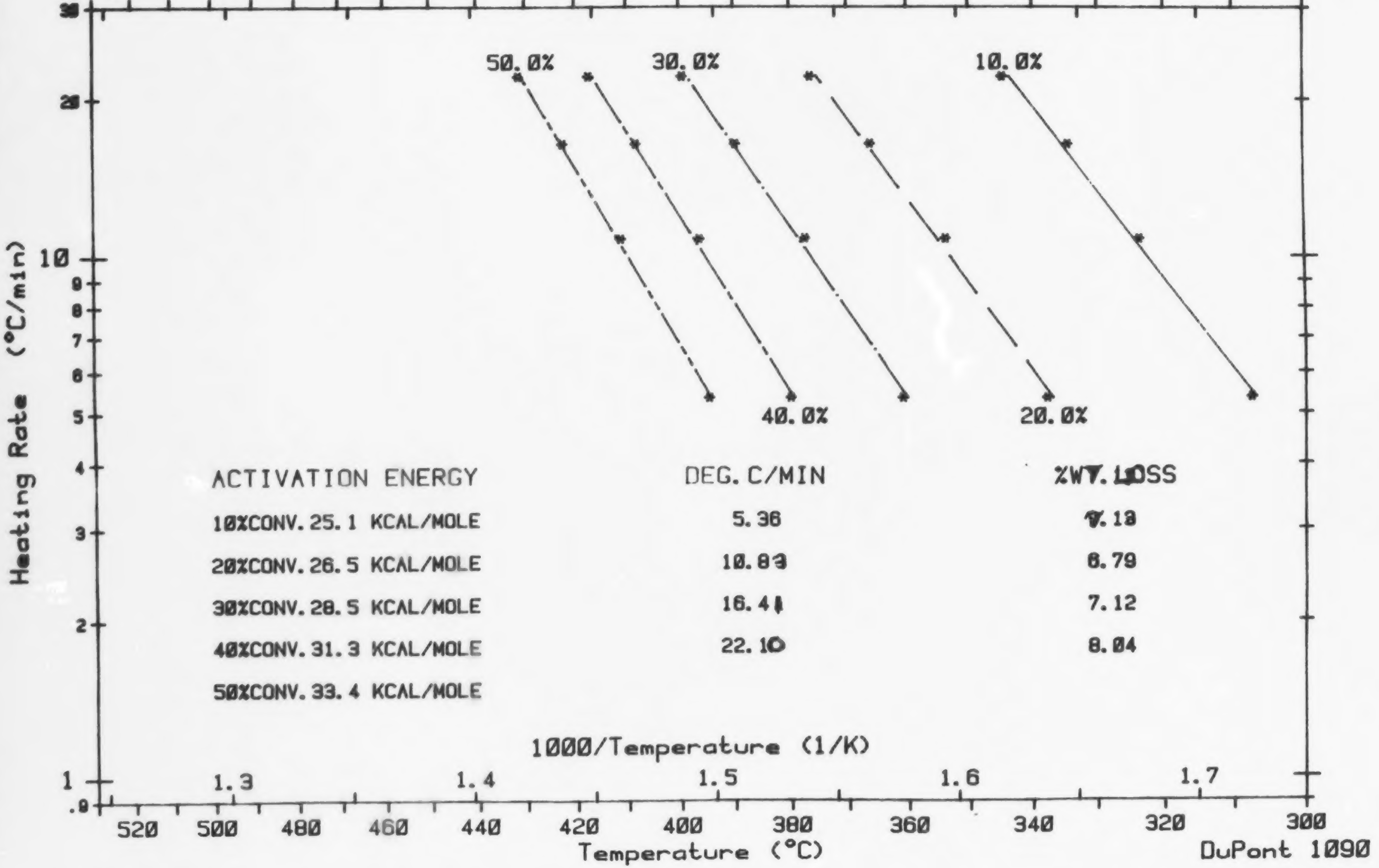
Sample: 45% ILLITE

Date: 27-Sep-85   Time: 14:56:55

Operator: JT

Program: TGA Kinetics V1.0

Plotted: 1-Oct-85   11:48:51

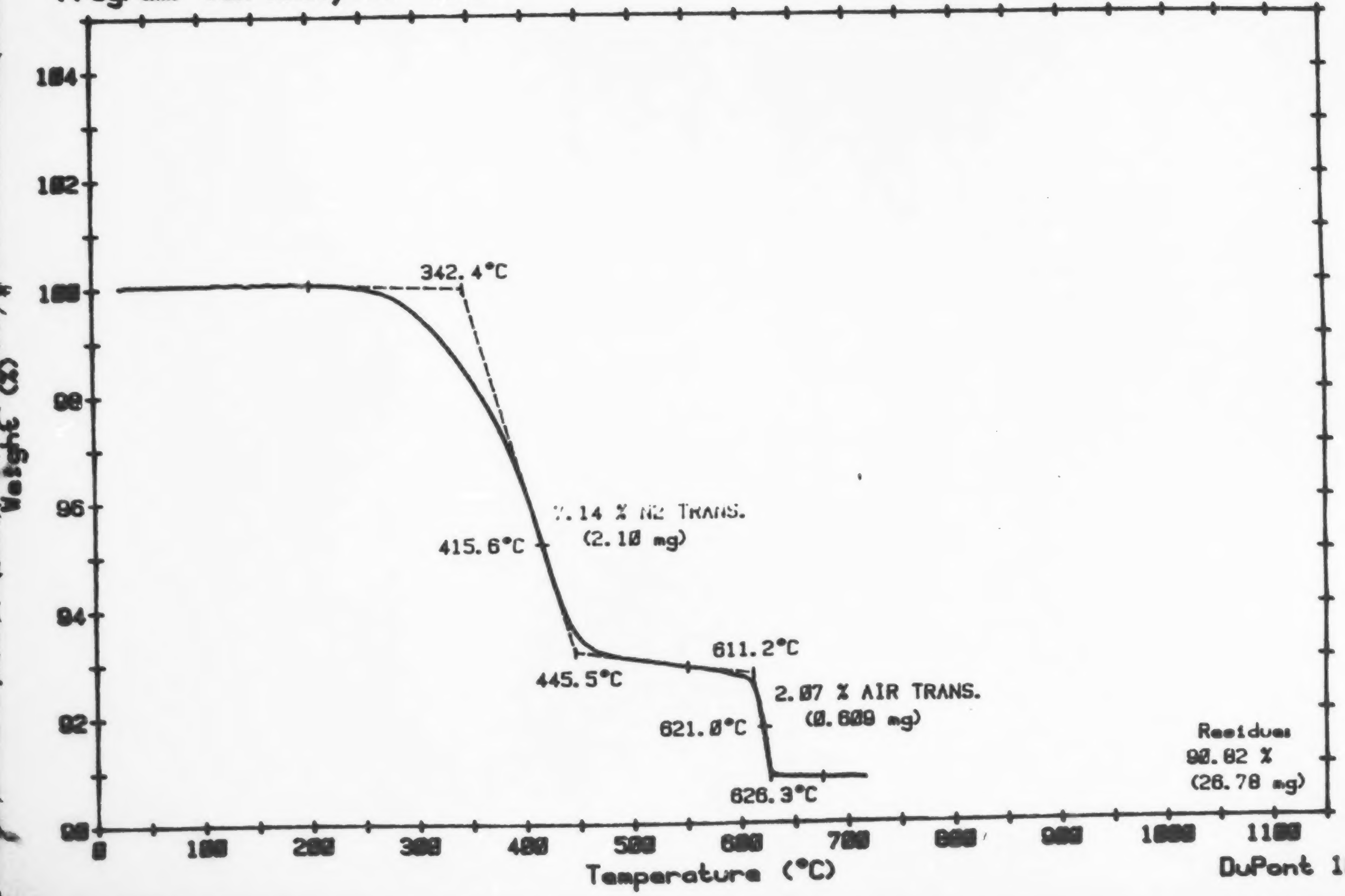


ACTIVATION ENERGY	DEG. C/MIN	%WT. LOSS
10%CONV. 25.1 KCAL/MOLE	5.36	7.18
20%CONV. 26.5 KCAL/MOLE	10.83	6.79
30%CONV. 28.5 KCAL/MOLE	16.41	7.12
40%CONV. 31.3 KCAL/MOLE	22.10	8.04
50%CONV. 33.4 KCAL/MOLE		

Sample: 45% ILLITE
Size: 29.49 mg
Rate: 05C/MIN
Program: TGA Analysis V2.0

TGA

Date: 26-Sep-85 Time: 16:06:58
File: PEL/RESID.10 T.LYNCH.00
Operator: JT
Plotted: 27-Sep-85 15:59:30



DuPont 1090

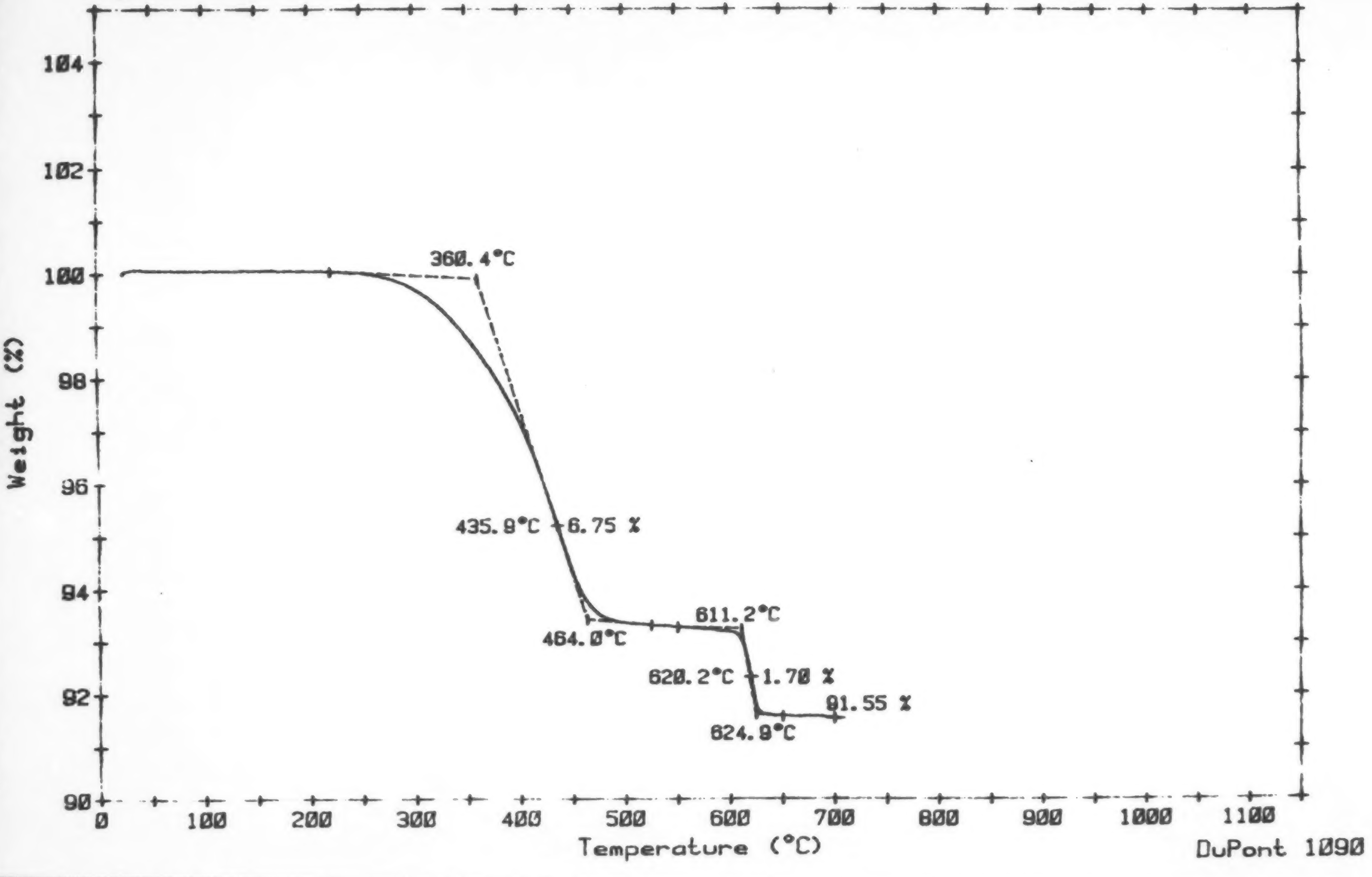
```
Sample: 45% ILLITE                                  Date: 26-Sep-85   Time: 16:06:58
Size: 29.49 mg                                      File: PEL/RESID.10 T.LYNCH.08
Rate: 05C/MIN                                       Operator: JT
Program: General Analysis V1.0                      Plotted: 30-Sep-85   13:31:13

     Temp      Sig-A     Deriv-A                         Temp      Sig-A     Deriv-A
     [deg]      [%]      [%/min]                         [deg]      [%]      [%/min]

     150.0    100.044      0.00                          250.2    99.959      0.01
     155.2    100.047      0.00                          255.1    99.942      0.02
     160.0    100.047      0.00                          260.1    99.919      0.01
     165.2    100.044      0.00                          265.1    99.888      0.01
     170.2    100.054      0.00                          270.0    99.854      0.02
     175.0    100.051      0.00                          275.1    99.810      0.03
     180.1    100.051      0.00                          280.2    99.769      0.03
     185.1    100.041      0.01                          285.0    99.708      0.03
     190.2    100.044      0.00                          290.2    99.630      0.02
     195.2    100.047      0.00                          295.1    99.559      0.05
     200.2    100.044      0.01                          300.2    99.468      0.05
     205.2    100.044      0.00                          305.1    99.373      0.06
     210.0    100.034      0.00                          310.0    99.281      0.05
     215.0    100.031      0.00                          315.2    99.186      0.05
     220.0    100.0[illegible]   [illegible]             320.1    99.064      0.06
     225.1    100.027      0.00                          325.2    98.932      0.08
     230.1    100.007      0.01                          330.1    98.810      0.08
     235.1    100.000      0.00                          335.2    98.688      0.07
     240.2     99.983      0.01                          340.1    98.545      0.08
     245.2     99.969      0.02                          345.2    98.399      0.09
                                                         350.0    98.253      0.09
```

```
Sample: 45% ILLITE                                Date: 26-Sep-85    Time: 16:06:58
Size: 29.49 mg                                    File: PEL/RESID.10 T.LYNCH.08
Rate: 05C/MIN                                     Operator: JT
Program: General Analysis V1.0                    Plotted: 30-Sep-85    13:44:39

      Temp       Sig-A      Deriv-A                    Temp       Sig-A      Deriv-A
      [deg]       [%]       [%/min]                    [deg]       [%]       [%/min]

      350.0     98.253       0.09                      450.2     93.441       0.05
      355.1     98.084       0.09                      455.1     93.336       0.04
      360.2     97.921       0.11                      460.2     93.261       0.03
      365.1     97.758       0.09                      465.1     93.204       0.03
      370.1     97.572       0.10                      470.1     93.156       0.02
      375.2     97.372       0.11                      475.0     93.139       0.02
      380.0     97.175       0.12                      480.0     93.109       0.02
      385.1     96.938       0.12                      485.1     93.088       0.01
      390.2     96.697       0.13                      490.2     93.064       0.01
      395.2     96.436       0.14                      495.2     93.058      -0.01
      400.0     96.168       0.16                      500.1     93.037       0.01
      405.1     95.866       0.17                      505.2     93.031       0.01
      410.0     95.547       0.17                      510.0     93.014       0.01
      415.1     95.204       0.18                      515.1     93.007       0.01
      420.1     94.8[illegible]  [illegible]           520.0     92.980       0.01
      425.0     94.547       0.16                      525.1     92.966       0.01
      430.0     94.258       0.15                      530.2     92.963       0.00
      435.0     93.990       0.13                      535.2     92.946       0.00
      440.2     93.750       0.11                      540.2     92.925      -0.01
      445.2     93.570       0.08                      545.2     92.919       0.01
                                                       550.1     92.908       0.00
```

Sample: 45% ILLITE
Size: 26.77 mg
Rate: 10C/MIN
Program: General Analysis V1.0

TGA

Date: 27-Sep-85 Time: 8:55:09
File: PEL/RESID.11 T.LYNCH.08
Operator: JT
Plotted: 30-Sep-85 14:34:50



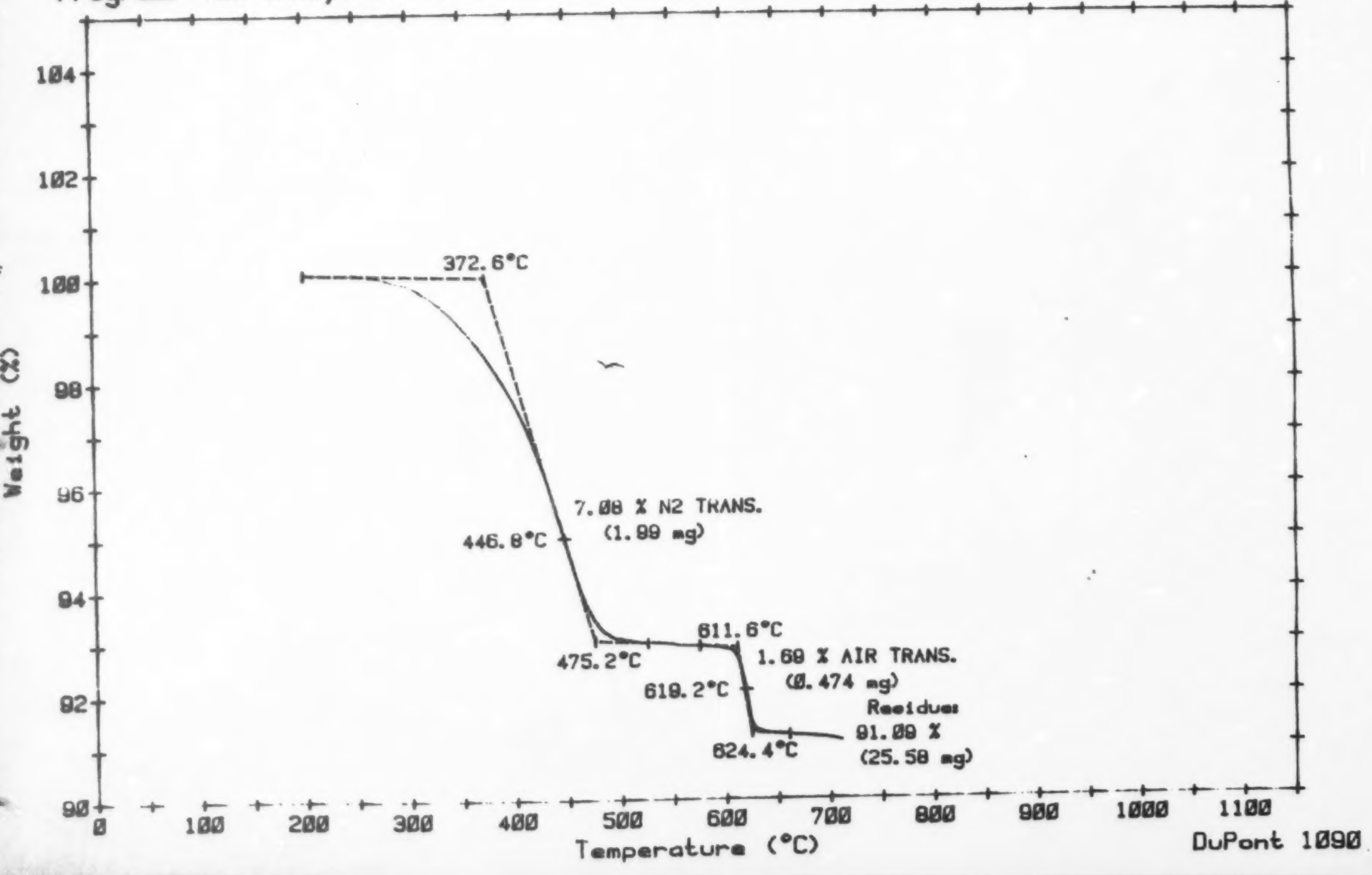
```
Sample: 45% ILLITE                                  Date: 27-Sep-85    Time:  8:55:09
Size: 26.77 mg                                      File: PEL/RESID.11 T.LYNCH.08
Rate: 10C/MIN                                       Operator: JT
Program: General Analysis V1.0                      Plotted: 30-Sep-85   15:24:01

       Temp       Sig-A       Deriv-A                       Temp       Sig-A       Deriv-A
      [deg]        [%]       [%/min]                       [deg]        [%]       [%/min]

      150.1     100.078        0.00                        250.5     100.030        0.01
      155.4     100.086        0.00                        255.0     100.018        0.02
      160.1     100.082        0.00                        260.0     100.015        0.00
      165.4     100.086        0.00                        265.1      99.985        0.02
      170.0     100.078        0.02                        270.1      99.966        0.04
      175.2     100.075       -0.01                        275.1      99.940        0.04
      180.5     100.075       -0.01                        280.1      99.910        0.04
      185.0     100.078        0.02                        285.1      99.877        0.03
      190.2     100.075       -0.01                        290.2      99.832        0.05
      195.3     100.078       -0.01                        295.2      99.787        0.05
      200.5     100.075        0.01                        300.1      99.735        0.07
      205.1     100.078        0.00                        305.1      99.668        0.06
      210.2     100.075       -0.01                        310.2      99.612        0.07
      215.4     100.075        0.00                        315.1      99.537        0.10
      220.5  [illegible]   [illegible]                     320.2      99.458        0.09
      225.1     100.063       -0.02                        325.1      99.373        0.10
      230.2     100.063        0.01                        330.0      99.275        0.10
      235.3     100.049        0.01                        335.5      99.163        0.12
      240.4     100.037       -0.01                        340.4      99.048        0.13
      245.4     100.048        0.01                        345.3      98.928        0.13
                                                           350.1      98.808        0.13
```

```
Sample: 45% ILLITE                                    Date: 27-Sep-85    Time:  8:55:09
Size: 26.77 mg                                        File: PEL/RESID.11 T.LYNCH.08
Rate: 10C/MIN                                         Operator: JT
Program: General Analysis V1.0                        Plotted: 30-Sep-85   15:36:35

     Temp       Sig-A      Deriv-A                      Temp       Sig-A      Deriv-A
     [deg]       [%]       [%/min]                      [deg]       [%]       [%/min]

     350.1     98.809       0.13                        450.0     94.349       0.28
     355.1     98.693       0.15                        455.2     94.095       0.23
     360.0     98.555       0.15                        460.5     93.882       0.20
     365.3     98.398       0.16                        465.2     93.740       0.14
     370.2     98.256       0.13                        470.3     93.621       0.10
     375.0     98.118       0.18                        475.5     93.520       0.06
     380.4     97.942       0.17                        480.2     93.464       0.05
     385.2     97.770       0.21                        485.3     93.426       0.03
     390.5     97.580       0.20                        490.5     93.400       0.03
     395.3     97.400       0.21                        495.1     93.378       0.02
     400.1     97.199       0.23                        500.2     93.363       0.01
     405.4     96.963       0.25                        505.4     93.344       0.03
     410.1     96.728       0.27                        510.0     93.341       0.01
     415.3     96.452       0.29                        515.1     93.341       0.00
     420.1     96.163       0.30                        520.1     93.326       0.01
     425.4     95.869       0.33                        525.2     93.322       0.02
     430.1     95.574       0.32                        530.2     93.318       0.00
     435.4     95.249       0.33                        535.3     93.314       0.01
     440.0     94.947       0.33                        540.3     93.292       0.01
     445.3     94.618       0.31                        545.3     93.288       0.01
                                                        550.3     93.285       0.01
```

Sample: 45% ILLITE
Size: 28.08 mg
Rate: 15C/MIN
Program: TGA Analysis V2.0

TGA

Date: 27-Sep-85 Time: 13:37:22
File: PEL/RESID.12 T.LYNCH.08
Operator: JT
Plotted: 27-Sep-85 14:32:36



Sample: 45% ILLITE
Size: 28.08 mg
Rate: 15C/MIN
Program: General Analysis V1.0

Date: 27-Sep-85 Time: 13:37:22
File: PEL/RESID.12 T.LYNCH.08
Operator: JT
Plotted: 1-Oct-85 8:23:02

Temp [deg]	Sig-A [%]	Deriv-A [%/min]
150.0	100.050	-0.01
155.3	100.053	0.00
160.7	100.057	0.01
165.2	100.061	0.00
170.5	100.061	0.00
175.8	100.064	0.01
180.1	100.061	0.00
185.4	100.061	0.00
190.7	100.050	0.01
195.1	100.053	0.00
200.3	100.053	-0.01
205.5	100.043	0.02
210.7	100.046	-0.01
215.1	100.050	0.00
220.3	[illegible]	[illegible]
225.5	100.043	0.02
230.7	100.032	0.01
235.0	100.028	0.01
240.1	100.028	-0.01
245.3	100.025	0.02

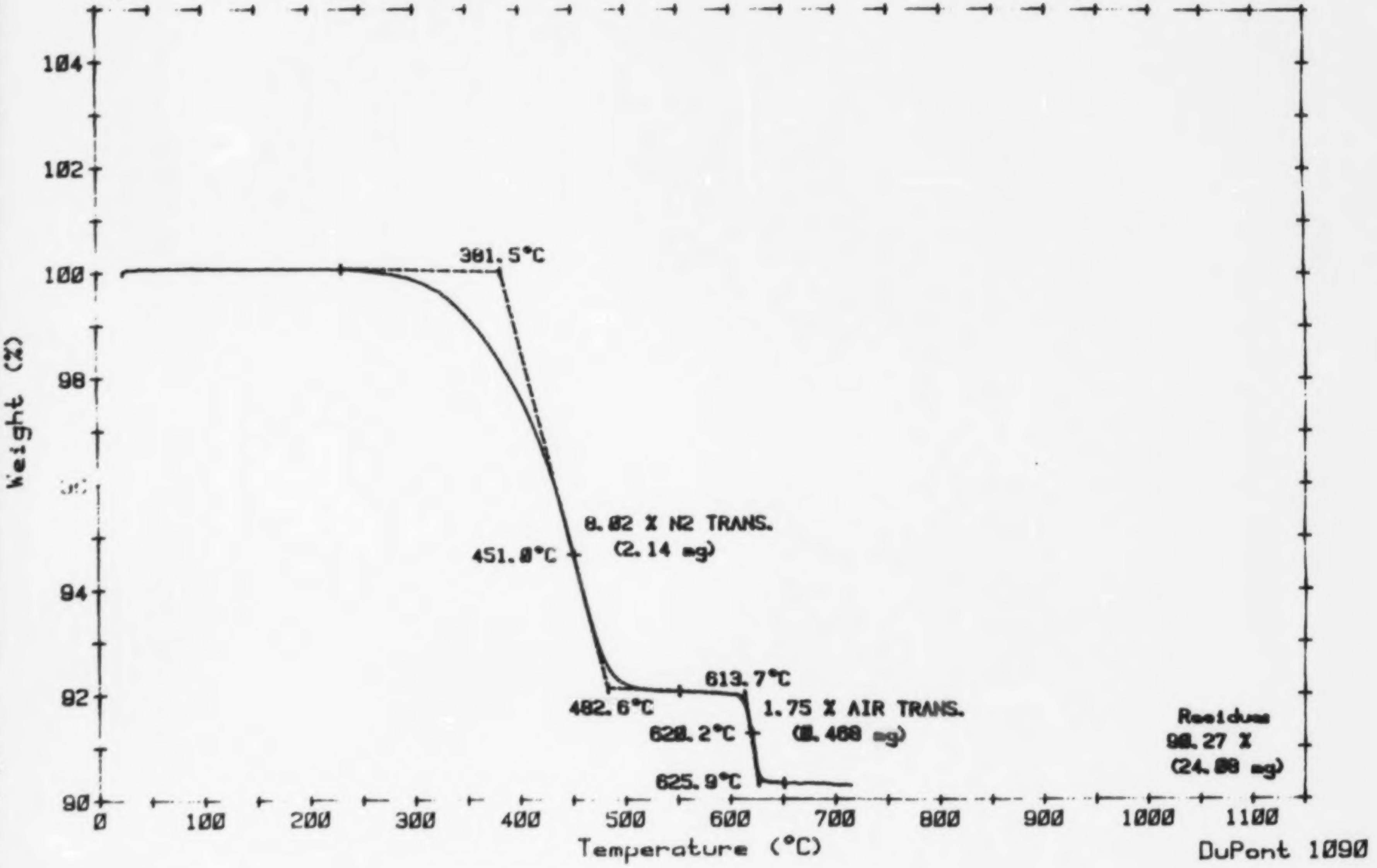
Temp [deg]	Sig-A [%]	Deriv-A [%/min]
250.5	100.011	0.02
255.6	100.007	0.00
260.7	100.000	0.01
265.8	99.989	0.03
270.0	99.968	0.04
275.0	99.950	0.04
280.0	99.918	0.04
285.0	99.893	0.05
290.1	99.872	0.03
295.2	99.843	0.05
300.2	99.808	0.07
305.2	99.765	0.08
310.1	99.697	0.11
315.0	99.637	0.11
320.0	99.573	0.11
325.0	99.498	0.13
330.1	99.420	0.14
335.7	99.320	0.14
340.6	99.220	0.18
345.6	99.113	0.19
350.6	98.989	0.18

```
Sample: 45% ILLITE                                        Date: 27-Sep-85    Time: 13:37:22
Size: 28.08 mg                                            File: PEL/RESID.12 T.LYNCH.08
Rate: 15C/MIN                                             Operator: JT
Program: General Analysis V1.0                            Plotted:  1-Oct-85     9:44:00

   Temp       Sig-A       Deriv-A                    Temp       Sig-A       Deriv-A
   [deg]      [%]         [%/min]                    [deg]      [%]         [%/min]

   350.6      98.999      0.18                       450.8      94.680      0.54
   355.5      98.889      0.20                       455.5      94.366      0.52
   360.5      98.764      0.21                       460.2      94.078      0.45
   365.4      98.640      0.21                       465.0      93.822      0.39
   370.3      98.511      0.23                       470.6      93.565      0.33
   375.1      98.362      0.24                       475.3      93.394      0.24
   380.0      98.212      0.25                       480.0      93.263      0.19
   385.7      98.034      0.26                       485.5      93.156      0.11
   390.6      97.881      0.27                       490.2      93.102      0.09
   395.5      97.710      0.28                       495.7      93.063      0.03
   400.3      97.536      0.33                       500.3      93.038      0.03
   405.1      97.336      0.34                       505.0      93.024      0.03
   410.1      97.119      0.36                       510.4      93.010      0.02
   415.6      96.849      0.41                       515.1      92.988      0.03
   420.4      96.606      0.42                       520.3      92.978      0.01
   425.3      96.343      0.45                       525.7      92.971      0.00
   430.1      96.062      0.48                       530.3      92.974      0.01
   435.7      95.716      0.51                       535.6      92.963      0.01
   440.4      95.381      0.53                       540.2      92.956      0.01
   445.2      95.075      0.55                       545.4      92.942      0.02
                                                     550.0      92.931      0.02
```

Sample: 45% ILLITE
Size: 26.68 mg
Rate: 20C/MIN
Program: TGA Analysis V2.0

TGA

Date: 27-Sep-85 Time: 14:56:55
File: PEL/RESID.13 T.LYNCH.08
Operator: JT
Plotted: 27-Sep-85 15:41:51



```
Sample: 45% ILLITE                                   Date: 27-Sep-85    Time: 14:56:55
Size: 26.68 mg                                       File: PEL/RESID.13 T.LYNCH.08
Rate: 20C/MIN                                        Operator: JT
Program: General Analysis V1.0                       Plotted:  3-Oct-85   10:08:59

       Temp        Sig-A       Deriv-A                       Temp       Sig-A      Deriv-A
       [deg]        [%]        [%/min]                       [deg]       [%]       [%/min]

       150.3      100.094        0.00                        250.5     100.075       0.03
       156.1      100.097       -0.02                        255.1     100.056       0.08
       160.8      100.097        0.01                        260.8     100.034       0.06
       165.5      100.097        0.00                        265.4     100.026       0.05
       170.3      100.097        0.00                        270.0     100.011       0.07
       175.0      100.097        0.03                        275.7      99.993       0.05
       180.8      100.086        0.04                        280.2      99.985       0.07
       185.5      100.079        0.00                        285.9      99.951       0.10
       190.1      100.090       -0.03                        290.4      99.936       0.11
       196.0      100.097        0.01                        296.1      99.910       0.13
       200.7      100.094       -0.02                        300.7      99.880       0.17
       205.4      100.097        0.00                        305.2      99.839       0.18
       210.1      100.094        0.01                        310.8      99.794       0.22
       215.9      100.090        0.03                        315.2      99.741       0.25
  [illegible]   [illegible]   [illegible]                    320.8      99.689       0.23
       225.1      100.082        0.00                        325.3      99.640       0.33
       230.8      100.079        0.00                        330.9      99.550       0.36
       235.5      100.062        0.02                        335.4      99.468       0.40
       240.1      100.071        0.04                        340.8      99.370       0.43
       245.9      100.067        0.00                        345.3      99.277       0.47
                                                             350.8      99.157       0.50
```

```
Sample: 45% ILLITE                                     Date: 27-Sep-85    Time: 14:56:55
Size: 26.58 mg                                         File: PEL/RESID.13 T.LYNCH.08
Rate: 20C/MIN                                          Operator: JT
Program: General Analysis V1.0                         Plotted:  3-Oct-85   10:34:16

      Temp        Sig-A      Deriv-A                        Temp        Sig-A      Deriv-A
      [deg]       [%]        [%/min]                        [deg]       [%]        [%/min]

      350.8       99.157       0.50                         451.0       94.640       1.70
      355.2       99.059       0.50                         455.2       94.288       1.70
      360.7       98.924       0.56                         460.5       93.876       1.64
      365.1       98.812       0.59                         465.8       93.482       1.49
      370.6       98.651       0.65                         470.1       93.197       1.35
      375.0       98.520       0.67                         475.4       92.883       1.16
      380.6       98.340       0.73                         480.6       92.624       0.87
      386.1       98.160       0.75                         485.9       92.444       0.63
      390.4       98.006       0.77                         490.1       92.332       0.46
      395.9       97.815       0.80                         495.4       92.242       0.29
      400.3       97.650       0.85                         500.6       92.189       0.17
      405.7       97.425       0.93                         505.8       92.148       0.14
      410.0       97.226       1.00                         511.0       92.118       0.10
      415.5       96.968       1.10                         515.2       92.107       0.05
      420.9       [illegible]  [illegible]                  520.4       92.092       0.07
      425.2       96.439       1.25                         525.5       92.084       0.07
      430.5       96.113       1.35                         530.6       92.066       0.03
      436.0       95.761       1.47                         535.7       92.062       0.02
      440.3       95.457       1.57                         540.7       92.058       0.00
      445.6       95.053       1.67                         545.8       92.054       0.01
                                                            551.0       92.054       0.03
```

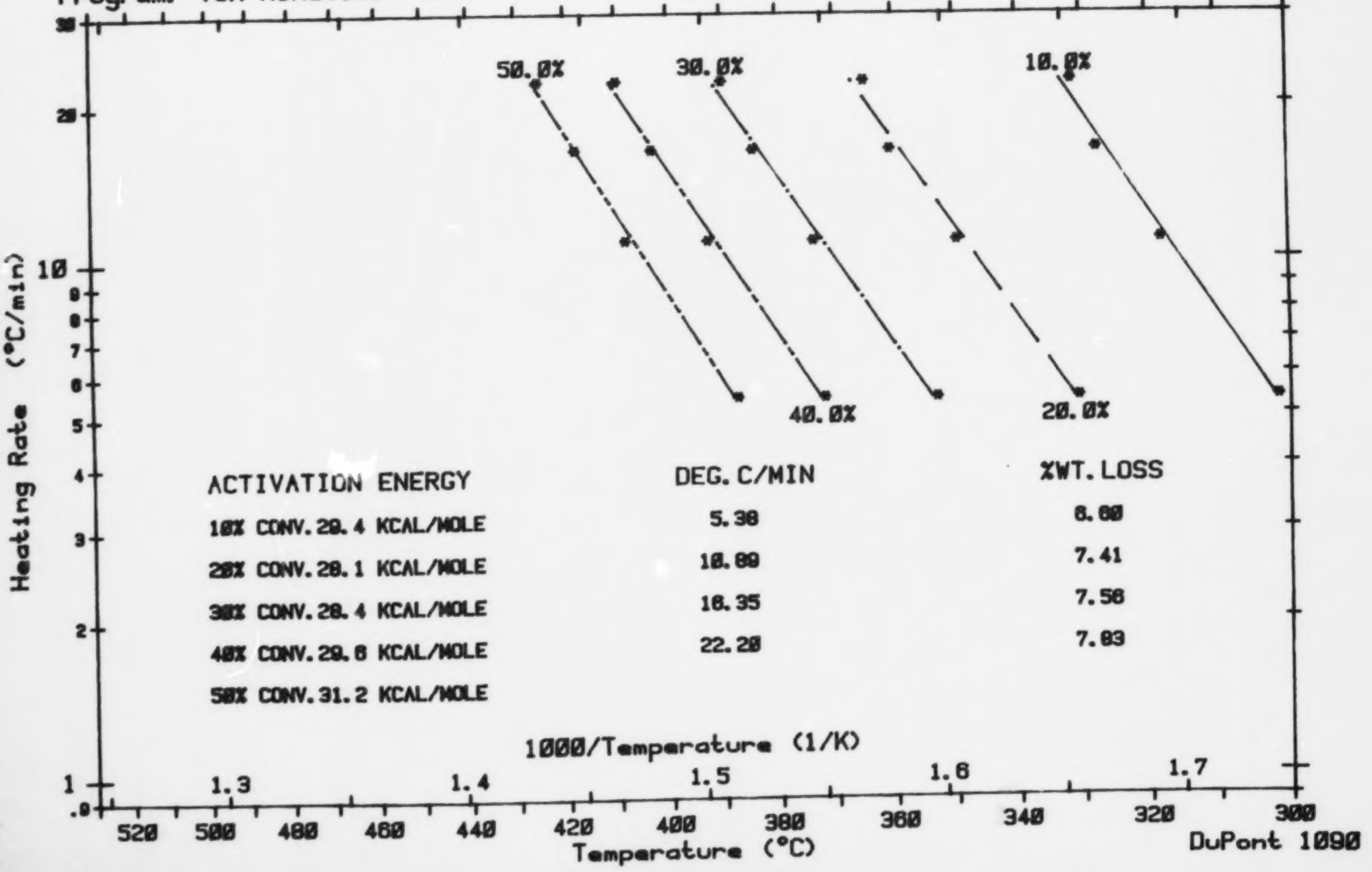
Sample: 90% ILLITE

Date: 3-Oct-85 Time: 9:56:09

Operator: JT

Program: TGA Kinetics V1.0

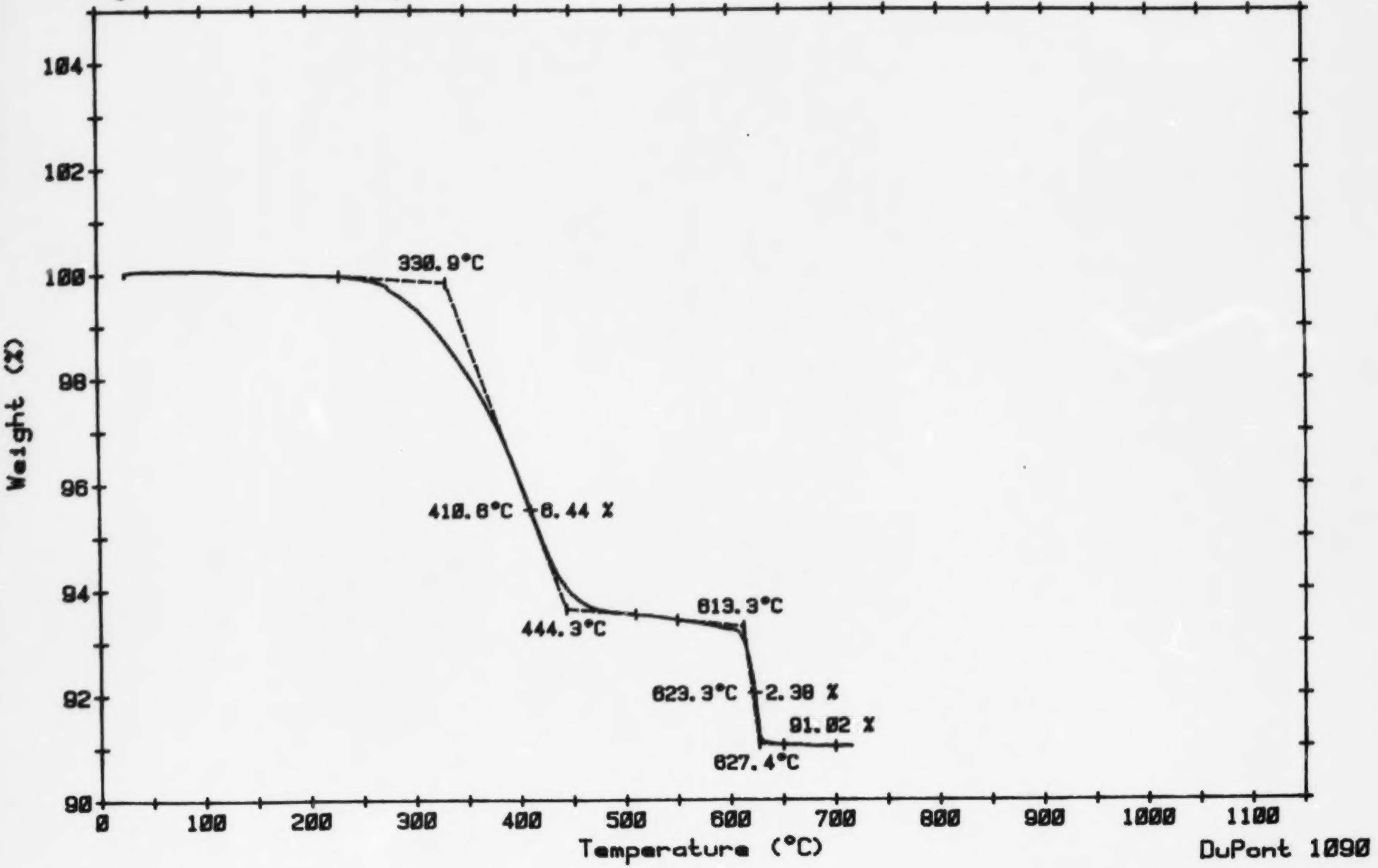
Plotted: 4-Oct-85 10:03:29



Sample: 90% ILLITE
Size: 26.53 mg
Rate: 05C/MIN
Program: General Analysis V1.0

TGA

Date: 3-Oct-85 Time: 15:37:35
File: PEL/RESID.43 T.LYNCH.08
Operator: JT
Plotted: 4-Oct-85 7:59:40



DuPont 1090

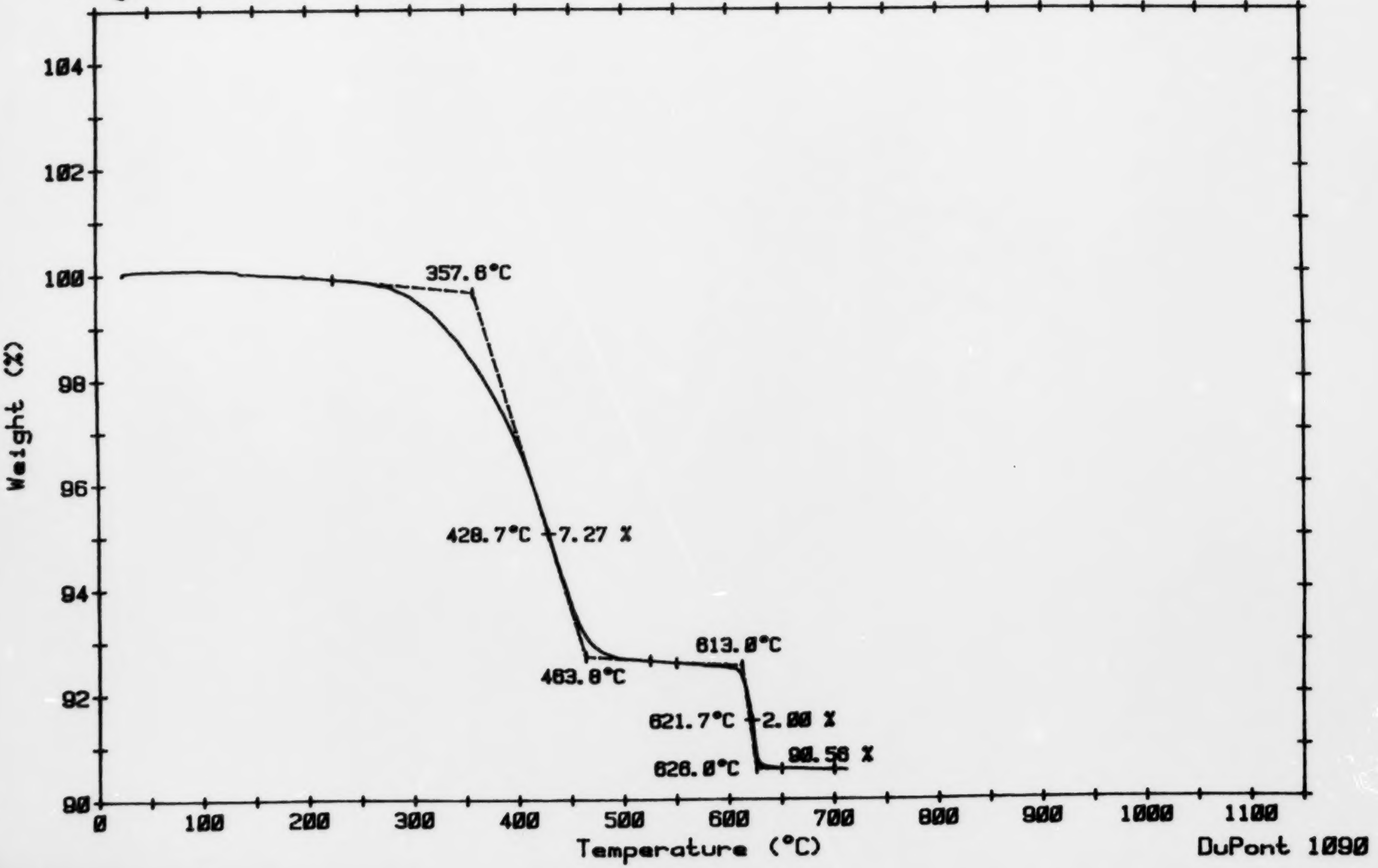
```
Sample: 90% ILLITE                                   Date:   3-Oct-85    Time: 15:37:35
Size: 26.53 mg                                       File: PEL/RESID.43 T.LYNCH.08
Rate: 05C/MIN                                        Operator: JT
Program: General Analysis V1.0                       Plotted: 10-Oct-85     8:26:27

     Temp      Sig-A      Deriv-A                     Temp      Sig-A      Deriv-A
     [deg]      [%]      [%/min]                      [deg]      [%]      [%/min]

     150.0    100.023      0.01                       250.1     99.925      0.02
     155.2    100.015      0.01                       255.1     99.906      0.02
     160.1    100.011      0.03                       260.1     99.887      0.03
     165.2    100.011      0.02                       265.2     99.857     -0.01
     170.0    100.000      0.00                       270.1     99.827      0.05
     175.2    100.004      0.01                       275.1     99.785      0.04
     180.1    100.008      0.00                       280.0     99.688      0.07
     185.1    100.004      0.01                       285.2     99.615      0.08
     190.2    100.000     -0.01                       290.2     99.551      0.08
     195.1     99.996      0.01                       295.1     99.476      0.09
     200.2     99.996      0.01                       300.0     99.387      0.10
     205.0     99.992      0.02                       305.2     99.289      0.13
     210.1     99.992      0.01                       310.2     99.189      0.12
     215.2     99.990      0.00                       315.1     99.084      0.13
     220.3     99.981      0.01                       320.3     98.963      0.12
     225.2     99.977      0.00                       325.2     98.846      0.14
     230.2     99.981      0.00                       330.1     98.711      0.14
     235.2     99.962      0.00                       335.2     98.571      0.15
     240.2     99.951      0.02                       340.1     98.435      0.15
     245.0     99.947      0.02                       345.2     98.281      0.17
                                                      350.1     98.130      0.14
```

```
Sample: 90% ILLITE                                  Date:   3-Oct-85    Time: 15:37:35
Size: 26.53 mg                                      File: PEL/RESID.43 T.LYNCH.08
Rate: 05C/MIN                                       Operator: JT
Program: General Analysis V1.0                      Plotted: 10-Oct-85     8:39:23

      Temp       Sig-A      Deriv-A                      Temp       Sig-A      Deriv-A
      [deg]       [%]       [%/min]                      [deg]       [%]       [%/min]

      350.1     98.130        0.14                       450.2     93.912        0.07
      355.2     97.968        0.18                       455.1     93.821        0.09
      360.1     97.810        0.21                       460.3     93.753        0.08
      365.2     97.629        0.21                       465.1     93.693        0.06
      370.2     97.440        0.19                       470.1     93.655        0.03
      375.1     97.252        0.23                       475.0     93.629        0.03
      380.2     97.041        0.23                       480.2     93.603        0.03
      385.0     96.830        0.23                       485.1     93.584        0.03
      390.0     96.584        0.26                       490.0     93.576        0.01
      395.1     96.336        0.27                       495.1     93.565        0.01
      400.1     96.076        0.29                       500.2     93.553        0.00
      405.2     95.800        0.29                       505.0     93.542        0.01
      410.0     95.540        0.28                       510.1     93.538        0.03
      415.0     95.281        0.28                       515.2     93.527        0.00
      420.1     94.997        0.25                       520.0     93.516        0.02
      425.1     94.741        0.25                       525.1     93.497        0.02
      430.0     94.511        0.24                       530.2     93.488        0.03
      435.2     94.307        0.19                       535.2     93.474        0.01
      440.0     94.157        0.14                       540.0     93.463        0.02
      445.0     94.025        0.13                       545.2     93.448        0.01
                                                         550.1     93.425        0.01
```

Sample: 90% ILLITE
Size: 26.45 mg
Rate: 10C/MIN
Program: General Analysis V1.0

TGA

Date: 4-Oct-85 Time: 8:26:08
File: PEL/RESID.44 T.LYNCH.08
Operator: JT
Plotted: 4-Oct-85 9:36:51



DuPont 1090

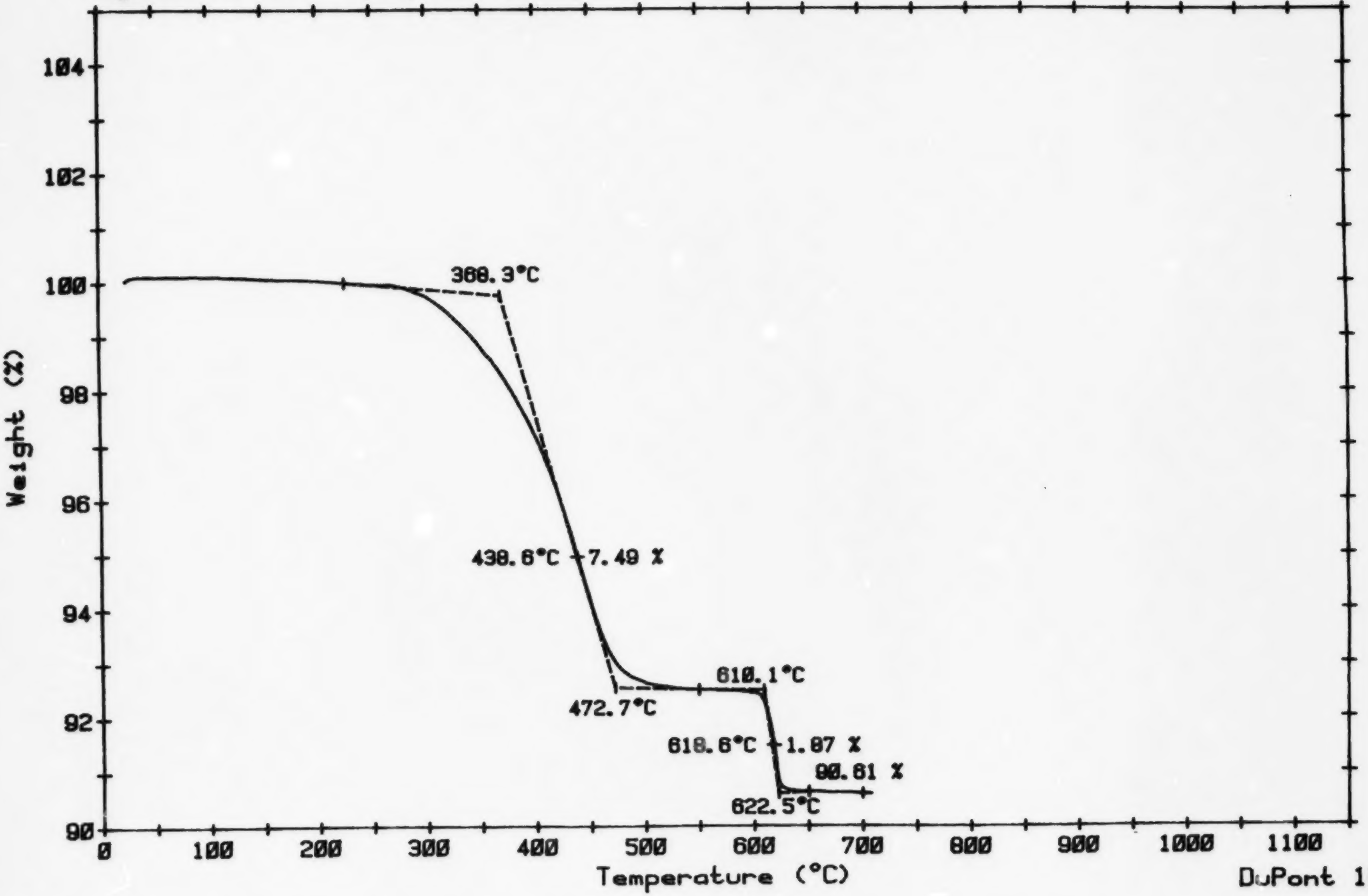
```
Sample: 90% ILLITE                                  Date:  4-Oct-85    Time:   8:26:08
Size:  26.45 mg                                     File:  PEL/RESID.44  T.LYNCH.08
Rate:  10C/MIN                                      Operator:  JT
Program:  General Analysis V1.0                     Plotted:  9-Oct-85    15:28:33

    Temp       Sig-A      Deriv-A                     Temp       Sig-A      Deriv-A
   [deg]        [%]       [%/min]                    [deg]        [%]       [%/min]

    150.1     99.998        0.02                     250.4     99.849        0.01
    155.2     99.992        0.00                     255.4     99.834        0.02
    160.4     99.985        0.02                     260.4     99.819        0.06
    165.6     99.977        0.02                     265.5     99.792        0.04
    170.1     99.974       -0.01                     270.5     99.773       -0.01
    175.4     99.977        0.02                     275.5     99.747        0.07
    180.5     99.970        0.01                     280.0     99.724        0.08
    185.2     99.962        0.03                     285.5     99.671        0.10
    190.3     99.955        0.01                     290.5     99.637        0.08
    195.4     99.955       -0.01                     295.0     99.595        0.11
    200.5     99.936       -0.01                     300.1     99.527        0.17
    205.0     99.932        0.03                     305.1     99.452        0.17
    210.2     99.924        0.01                     310.1     99.369        0.16
    215.4     99.921        0.01                     315.1     99.297        0.18
    220.4     99.905        0.02                     320.2     99.214        0.24
    225.0     99.898        0.02                     325.2     99.096        0.23
    230.0     99.894        0.03                     330.2     98.990        0.25
    235.2     99.879        0.01                     335.1     98.869        0.26
    240.4     99.875        0.01                     340.1     98.771        0.28
    245.4     99.864        0.03                     345.1     98.643        0.31
                                                     350.4     98.491        0.33
```

```
Sample: 90% ILLITE                                 Date:   4-Oct-85    Time:   8:26:08
Size:  26.45 mg                                    File:  PEL/RESID.44 T.LYNCH.08
Rate:  10C/MIN                                     Operator:  JT
Program:  General Analysis V1.0                    Plotted:   9-Oct-85    15:37:32

     Temp       Sig-A      Deriv-A                      Temp       Sig-A      Deriv-A
     [deg]       [%]       [%/min]                      [deg]       [%]       [%/min]

     350.4     98.491       0.33                        450.0     93.708       0.61
     355.3     98.344       0.24                        455.2     93.436       0.50
     360.2     98.215       0.34                        460.5     93.209       0.37
     365.0     98.057       0.36                        465.2     93.058       0.31
     370.0     97.890       0.37                        470.5     92.933       0.22
     375.0     97.720       0.41                        475.1     92.858       0.16
     380.4     97.523       0.42                        480.3     92.797       0.09
     385.3     97.346       0.41                        485.0     92.756       0.08
     390.1     97.142       0.46                        490.2     92.718       0.08
     395.4     96.911       0.48                        495.4     92.688       0.06
     400.3     96.684       0.52                        500.4     92.669       0.02
     405.2     96.446       0.52                        505.1     92.665       0.04
     410.0     96.177       0.61                        510.1     92.650       0.02
     415.4     95.871       0.63                        515.2     92.650       0.03
     420.2     95.588       0.68                        520.2     92.635       0.01
     425.0     95.278       0.69                        525.2     92.631       0.02
     430.3     94.926       0.69                        530.1     92.619       0.01
     435.1     94.612       0.72                        535.2     92.616       0.04
     440.4     94.249       0.65                        540.2     92.597       0.02
     445.3     93.988       0.69                        545.2     92.589       0.03
                                                        550.3     92.582       0.01
```

Sample: 90% ILLITE
Size: 26.67 mg
Rate: 15C/MIN
Program: General Analysis V1.0

TGA

Date: 3-Oct-85 Time: 12:22:41
File: PEL/RESID.41 T.LYNCH.08
Operator: JT
Plotted: 3-Oct-85 13:33:23



DuPont 1090

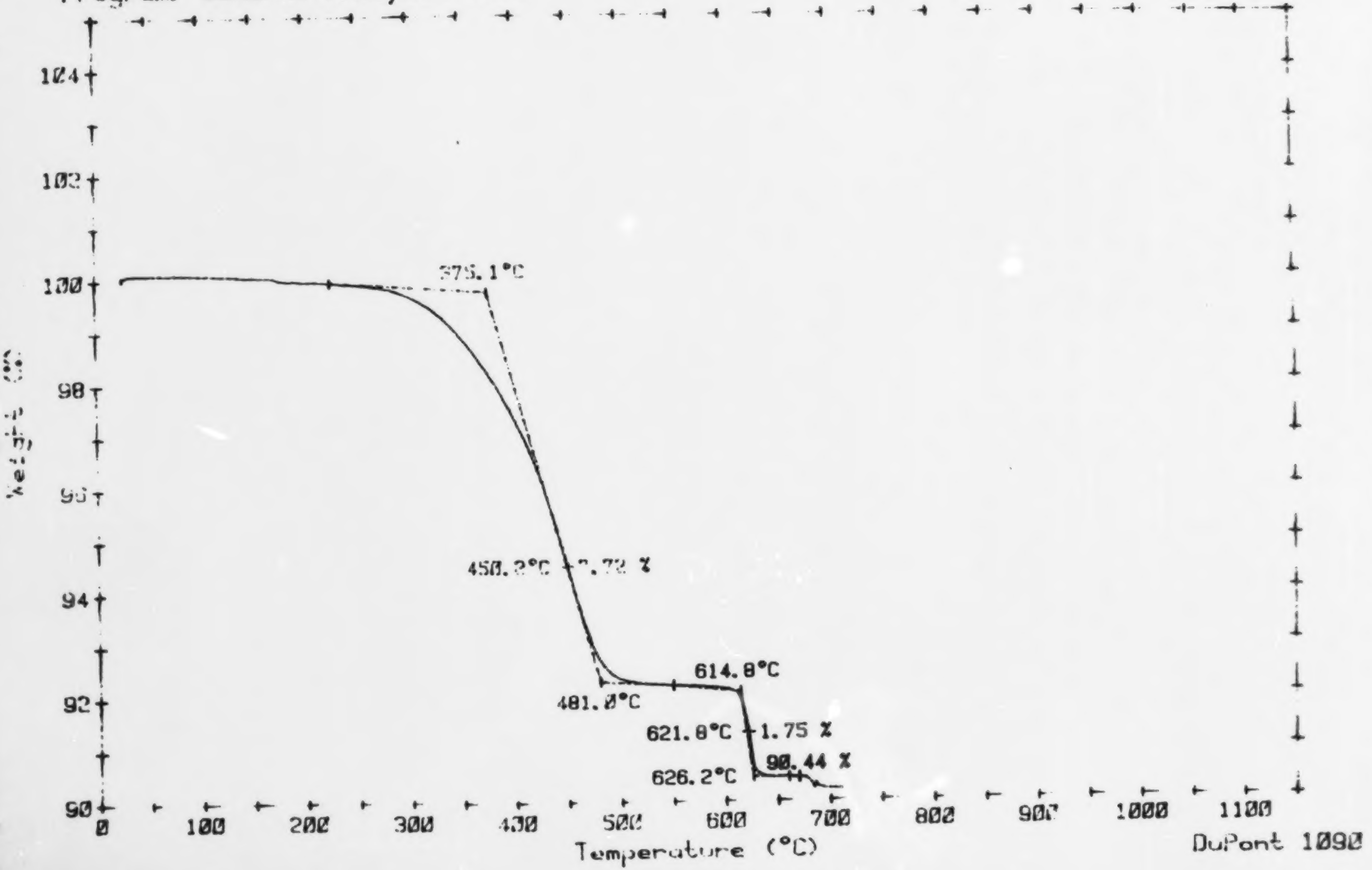
```
Sample: 90% ILLITE                                   Date:   3-Oct-85    Time: 12:22:41
Size:  26.67 mg                                      File:  PEL/RESID.41  T.LYNCH.08
Rate:  15C/MIN                                       Operator:  JT
Program:  General Analysis V1.0                      Plotted:  10-Oct-85     8:49:45

    Temp        Sig-A       Deriv-A                  Temp       Sig-A       Deriv-A
    [deg]        [%]        [%/min]                  [deg]       [%]        [%/min]

    150.7      100.075        0.02                   250.2      99.974        0.01
    155.2      100.071        0.01                   255.3      99.970       -0.01
    160.5      100.067        0.03                   260.4      99.966        0.01
    165.6      100.064        0.00                   265.5      99.970        0.02
    170.1      100.060        0.01                   270.5      99.959        0.05
    175.4      100.060        0.00                   275.6      99.933        0.09
    180.5      100.056        0.01                   280.6      99.906        0.10
    185.7      100.056        0.00                   285.6      99.873        0.12
    190.6      100.056        0.01                   290.6      99.831        0.11
    195.0      100.049        0.02                   295.6      99.798        0.14
    200.1      100.049        0.01                   300.6      99.749        0.19
    205.1      100.041        0.02                   305.6      99.685        0.22
    210.3      100.030        0.05                   310.5      99.618        0.25
    215.4      100.022        0.02                   315.5      99.539        0.28
    220.5      100.015        0.01                   320.5      99.456        0.31
    225.6      100.007        0.05                   325.4      99.363        0.32
    230.6       99.996        0.01                   330.4      99.265        0.34
    235.7       99.992        0.00                   335.4      99.164        0.33
    240.0       99.989        0.04                   340.4      99.052        0.38
    245.1       99.974        0.02                   345.3      98.935        0.41
                                                     350.3      98.808        0.43
```

```
Sample: 90% ILLITE                                  Date:   3-Oct-85    Time: 12:22:41
Size: 26.67 mg                                      File: PEL/RESID.41 T.LYNCH.08
Rate: 15C/MIN                                       Operator: JT
Program: General Analysis V1.0                      Plotted: 10-Oct-85    9:03:01

     Temp       Sig-A       Deriv-A                      Temp       Sig-A       Deriv-A
     [deg]       [%]        [%/min]                      [deg]       [%]        [%/min]

     350.3      98.808        0.43                       450.3      94.137        1.03
     355.2      98.680        0.34                       455.0      93.829        0.96
     360.1      98.575        0.46                       460.6      93.518        0.82
     365.1      98.433        0.49                       465.3      93.289        0.69
     370.0      98.283        0.50                       470.1      93.106        0.52
     375.8      98.096        0.54                       475.6      92.948        0.38
     380.6      97.934        0.56                       480.3      92.854        0.26
     385.5      97.758        0.61                       485.7      92.772        0.20
     390.3      97.582        0.59                       490.4      92.727        0.12
     395.2      97.391        0.67                       495.1      92.690        0.12
     400.1      97.185        0.71                       500.5      92.637        0.16
     405.8      96.926        0.76                       505.0      92.615        0.06
     410.6      96.692        0.84                       510.4      92.596        0.04
     415.4      96.431        0.88                       515.0      92.581        0.03
     420.3      96.157        0.94                       520.4      92.562        0.06
     425.1      95.865        1.00                       525.7      92.551        0.04
     430.6      95.497        1.07                       530.3      92.536        0.03
     435.4      95.171        1.11                       535.6      92.532        0.03
     440.1      94.845        1.13                       540.2      92.521        0.02
     445.7      94.448        1.08                       545.4      92.517        0.01
                                                         550.0      92.513        0.00
```

Sample: 90% ILLITE
Size: 27.52 mg
Rate: 20C/MIN
Program: General Analysis V1.0

TGA

Date: 3-Oct-85 Time: 9:56:09
File: PEL/RESID.40 T.LYNCH.08
Operator: JT
Plotted: 3-Oct-85 10:45:25



DuPont 1090

```
Sample: 90% ILLITE                                  Date:   3-Oct-85    Time:   9:56:09
Size: 27.52 mg                                      File: PEL/RESID.40 T.LYNCH.08
Rate: 20C/MIN                                       Operator: JT
Program: General Analysis V1.0                      Plotted: 10-Oct-85    9:48:48

    Temp       Sig-A      Deriv-A                    Temp       Sig-A      Deriv-A
    [deg]       [%]       [%/min]                    [deg]       [%]       [%/min]

    151.1     100.036       0.03                     250.8      99.880       0.06
    155.9     100.029       0.03                     255.5      99.869       0.04
    160.6     100.025       0.02                     260.1      99.862       0.05
    165.3     100.018       0.02                     265.8      99.847       0.07
    170.1     100.018       0.03                     270.4      99.833       0.08
    176.2      99.982       0.16                     275.0      99.815       0.09
    180.9      99.967       0.04                     280.7      99.793       0.11
    185.6      99.964       0.02                     285.3      99.767       0.13
    190.4      99.956       0.02                     291.0      99.727       0.17
    195.1      99.953       0.03                     295.5      99.695       0.18
    201.0      99.945       0.01                     300.1      99.655       0.20
    205.6      99.949       0.01                     305.8      99.608       0.24
    210.3      99.942       0.02                     310.3      99.557       0.28
    216.1      99.938       0.03                     315.9      99.488       0.29
    220.8      99.931       0.03                     320.4      99.426       0.32
    225.5      99.927       0.02                     326.0      99.339       0.36
    230.1      99.920       0.03                     330.5      99.266       0.39
    235.9      99.913       0.05                     336.0      99.181       0.46
    240.5      99.898       0.04                     340.5      99.068       0.48
    245.1      99.895       0.05                     346.1      98.943       0.51
                                                     350.6      98.837       0.52
```

```
Sample: 90% ILLITE                                      Date:   3-Oct-85    Time:   9:56:09
Size: 27.52 mg                                          File: PEL/RESID.40 T.LYNCH.08
Rate: 20C/MIN                                           Operator: JT
Program: General Analysis V1.0                          Plotted: 10-Oct-85     9:57:45

      Temp       Sig-A      Deriv-A                        Temp       Sig-A      Deriv-A
      [deg]       [%]       [%/min]                        [deg]       [%]       [%/min]

      350.6     98.837        0.52                         450.2     94.484        1.53
      355.1     98.732        0.57                         455.5     94.103        1.51
      360.6     98.583        0.60                         460.9     93.732        1.43
      365.1     98.459        0.62                         465.1     93.456        1.33
      370.5     98.300        0.63                         470.5     93.140        1.18
      375.0     98.176        0.67                         475.8     92.878        0.91
      380.5     98.002        0.70                         480.0     92.719        0.73
      386.0     97.816        0.76                         485.2     92.559        0.52
      390.4     97.684        0.79                         490.5     92.453        0.35
      396.0     97.460        0.84                         495.8     92.384        0.23
      400.4     97.289        0.87                         500.0     92.341        0.18
      405.8     97.088        0.92                         505.2     92.304        0.11
      410.2     96.879        0.98                         510.4     92.283        0.08
      415.7     96.625        1.05                         515.6     92.264        0.08
      420.0     96.407        1.13                         520.7     92.246        0.07
      425.4     96.116        1.21                         525.8     92.232        0.02
      430.9     95.800        1.33                         530.0     92.232        0.03
      435.1     95.524        1.39                         535.1     92.221        0.02
      440.5     95.167        1.47                         540.2     92.213        0.02
      445.9     94.790        1.52                         545.3     92.213        0.02
                                                           550.4     92.203        0.04
```

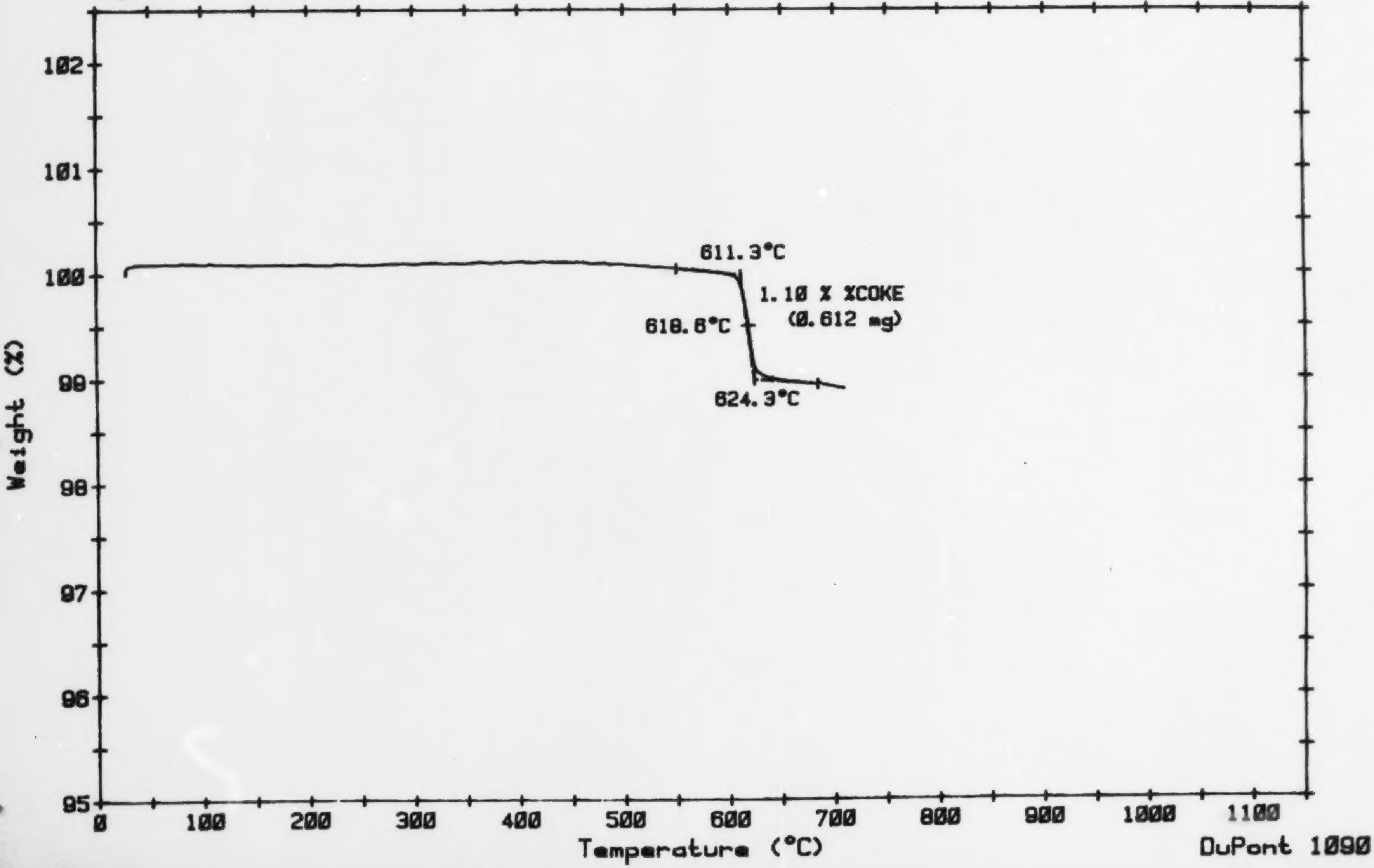
APPENDIX B

TGA DATA FOR COKE DEVOLATILIZATION

Sample: RUN#52
Size: 55.77 mg
Rate: 15C/MIN
Program: TGA Analysis V2.0

TGA

Date: 9-Oct-85 Time: 14:56:24
File: COKE.01 T.LYNCH.08
Operator: JT
Plotted: 10-Oct-85 8:16:49



DuPont 1090

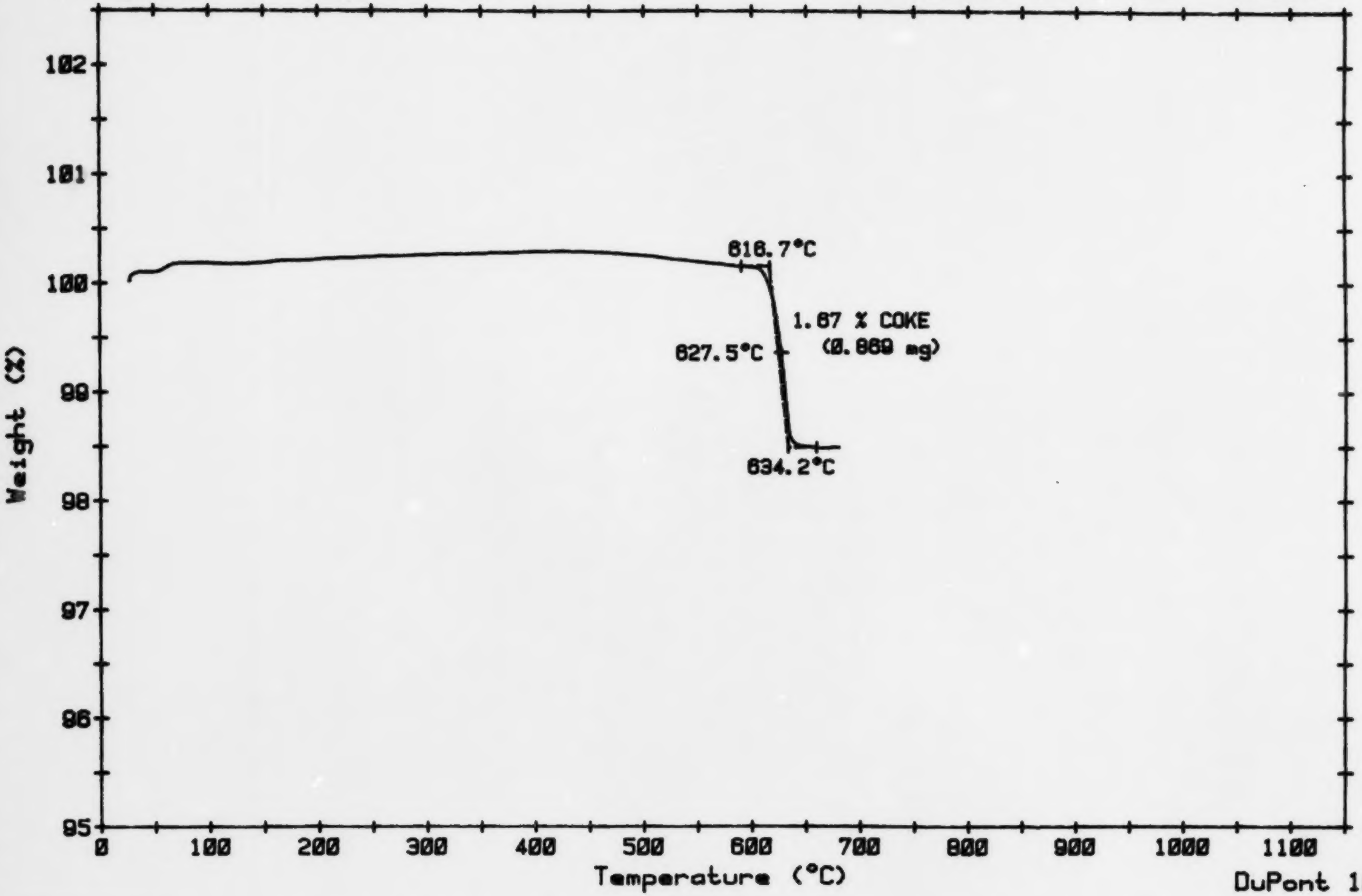
```
Sample: RUN#52                                Date:   9-Oct-85    Time: 14:56:24
Size: 55.77 mg                                File: COKE.01 T.LYNCH.08
Rate: 15C/MIN                                 Operator: JT
Program: General Analysis V1.0                Plotted: 29-Oct-85     8:47:06

      Temp        Sig-A      Deriv-A                Temp       Sig-A      Deriv-A
      [deg]        [%]       [%/min]                [deg]       [%]       [%/min]

  [illegible]   100.100         0.00               475.5     100.086         0.01
      405.8     100.100         0.00               480.3     100.082        -0.01
      410.7     100.099         0.00               485.1     100.081         0.01
      415.7     100.104        -0.01               490.5     100.077         0.01
      420.5     100.097        -0.01               495.3     100.077         0.01
      425.5     100.099         0.02               500.0     100.072         0.01
      430.5     100.102        -0.01               505.5     100.070         0.01
      435.4     100.102         0.01               510.1     100.065         0.01
      440.2     100.097         0.00               515.5     100.058         0.00
      445.1     100.097         0.01               520.2     100.057         0.02
      450.7     100.090         0.00               525.5     100.048         0.03
      455.5     100.097         0.00               530.1     100.052         0.01
      460.3     100.097         0.00               535.8     100.048         0.02
      465.1     100.090         0.03               540.1     100.041         0.00
      470.7     100.088         0.00               545.4     100.039         0.02
                                                   550.1     100.032         0.01
```

```
Sample: RUN#52                                Date:   9-Oct-85   Time: 14:56:24
Size: 55.77 mg                                File: COKE.01 T.LYNCH.08
Rate: 15C/MIN                                 Operator: JT
Program: General Analysis V1.0                Plotted: 29-Oct-85     8:54:18

    Temp      Sig-A     Deriv-A                   Temp      Sig-A     Deriv-A
    [deg]      [%]      [%/min]                   [deg]      [%]      [%/min]

    550.1   100.032       0.01                    625.2    99.082       0.20
    555.4   100.032       0.02                    630.4    99.030       0.06
    560.0   100.022       0.01                    635.0    99.001       0.01
    565.2   100.025       0.00                    640.2    98.994       0.02
    570.3   100.016       0.02                    645.5    98.976       0.01
    575.7   100.016       0.02                    650.2    98.971       0.03
    580.2   100.013       0.02                    655.4    98.958       0.02
    585.4   100.004       0.00                    660.1    98.953       0.01
    590.5   100.002       0.02                    665.3    98.956       0.01
    595.2    99.986       0.03                    670.4    98.947       0.02
    600.3    99.978       0.00                    675.2    98.944       0.01
    605.1    99.964       0.07                    680.3    98.940       0.01
    610.2    99.900       0.25                    685.0    98.935       0.02
    615.2    99.717       0.61                    690.1    98.929       0.04
    620.2    99.36[illegible]  [illegible]        695.2    98.917       0.01
                                                  700.3    98.906       0.03
```

Sample: RUN#54
Size: 52.11 mg
Rate: 15C/MIN
Program: TGA Analysis V2.0

TGA

Date: 11-Oct-85 Time: 15:38:47
File: COKE.10 T.LYNCH.08
Operator: JT
Plotted: 15-Oct-85 8:00:23



DuPont 1090

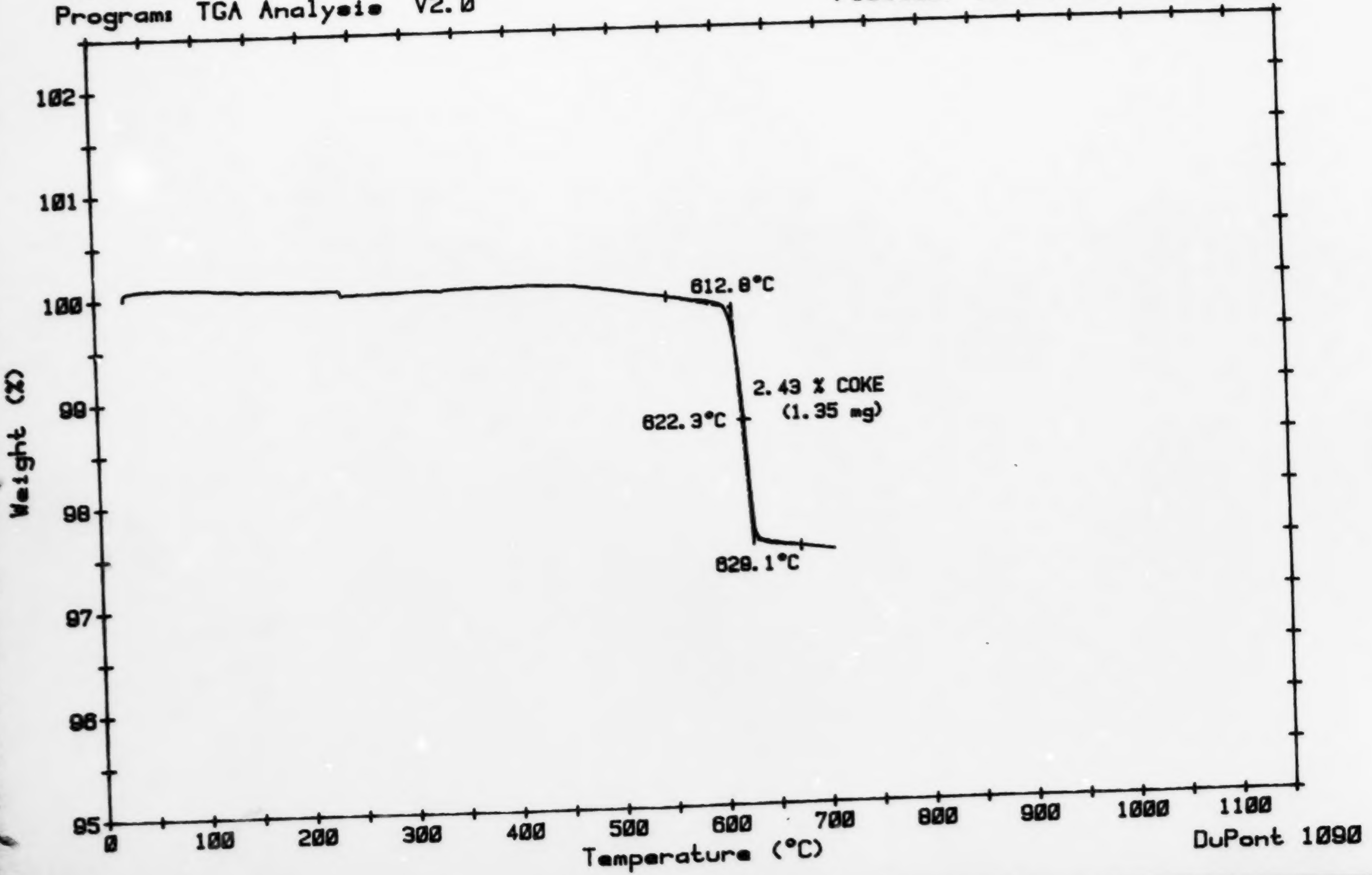
```
Sample: RUN#54                                     Date: 11-Oct-85    Time: 15:38:47
Size: 52.11 mg                                     File: COKE.10 T.LYNCH.08
Rate: 15C/MIN                                      Operator: JT
Program: General Analysis V1.0                     Plotted: 1-Nov-85    15:20:36

     Temp      Sig-A     Deriv-A                        Temp      Sig-A     Deriv-A
     [deg]     [%]       [%/min]                        [deg]     [%]       [%/min]

     400.4     100.297      0.00                        475.1     100.280      0.01
     405.6     100.297     -0.01                        480.2     100.278      0.01
     410.7     100.297      0.00                        485.2     100.272      0.02
     415.8     100.297      0.00                        490.4     100.267      0.02
     420.1     100.297      0.00                        495.5     100.265      0.01
     425.3     100.299      0.00                        500.4     100.261      0.00
     430.4     100.296      0.00                        505.3     100.257      0.02
     435.5     100.294      0.01                        510.3     100.253      0.02
     440.6     100.296      0.00                        515.2     100.246      0.02
     445.7     100.294      0.00                        520.1     100.238      0.02
     450.0     100.292      0.00                        525.2     100.232      0.03
     455.1     100.290      0.01                        530.1     100.225      0.01
     460.2     100.286      0.01                        535.0     100.219      0.03
     465.2     100.288      0.01                        540.7     100.215      0.01
     470.1     100.284      0.01                        545.6     100.211      0.02
                                                        550.6     100.203      0.02
```

```
Sample: RUN#54                                     Date: 11-Oct-85    Time: 15:38:47
Size: 52.11 mg                                     File: COKE.10 T.LYNCH.08
Rate: 15C/MIN                                      Operator: JT
Program: General Analysis V1.0                     Plotted: 1-Nov-85    15:29:28

      Temp        Sig-A      Deriv-A                   Temp       Sig-A      Deriv-A
      [deg]        [%]      [%/min]                    [deg]       [%]       [%/min]

      550.6      100.203       0.02                    616.0      99.990       0.65
      555.5      100.200       0.01                    620.5      99.822       1.11
      560.4      100.192       0.02                    625.3      99.541       1.59
      565.3      100.188       0.01                    630.2      99.115       1.54
      570.2      100.186       0.01                    635.1      98.622       0.42
      575.6      100.178       0.02                    640.0      98.536       0.10
      580.7      100.173       0.02                    645.0      98.513       0.04
      585.5      100.165       0.01                    650.3      98.505       0.01
      590.3      100.165       0.00                    655.7      98.499       0.01
      595.0      100.161       0.02                    660.3      98.497       0.01
      600.6      100.154       0.01                    665.7      98.490       0.01
      605.8      100.148       0.10                    670.3      98.490      -0.01
      610.7      100.100       0.29                    675.1      98.499      -0.02
                                                       680.7      98.494       0.00
```

Sample: RUN#54
Size: 55.31 mg
Rate: 15C/MIN
Program: TGA Analysis V2.0

TGA

Date: 10-Oct-85 Time: 10:09:52
File: COKE.02 T.LYNCH.08
Operator: JT
Plotted: 10-Oct-85 11:01:44



DuPont 1090

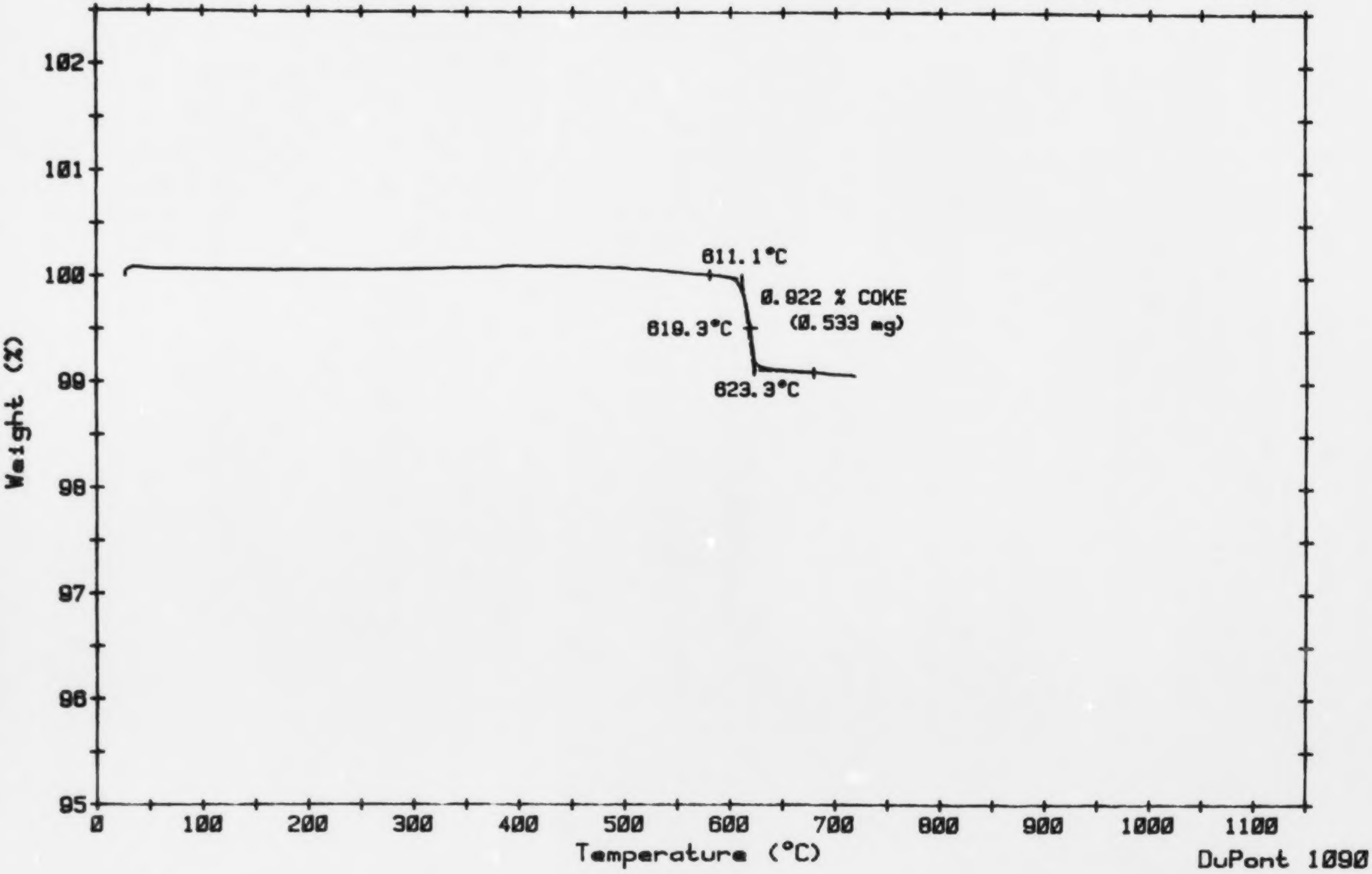
```
Sample: RUN#54                                        Date: 10-Oct-85    Time: 10:09:52
Size: 55.31 mg                                        File: COKE.02 T.LYNCH.08
Rate: 15C/MIN                                         Operator: JT
Program: General Analysis V1.0                        Plotted: 1-Nov-85    14:54:37

      Temp       Sig-A      Deriv-A                   Temp       Sig-A      Deriv-A
      [deg]       [%]       [%/min]                   [deg]       [%]       [%/min]

      400.6     100.042      -0.01                    475.1     100.011       0.02
      405.6     100.045       0.00                    480.6     100.002       0.01
      410.5     100.043      -0.01                    485.3     100.002       0.02
      415.5     100.049       0.00                    490.1      99.993       0.03
      420.3     100.051       0.01                    495.6      99.980       0.02
      425.3     100.045       0.01                    500.4      99.975       0.02
      430.1     100.042       0.01                    505.1      99.971       0.03
      435.7     100.038       0.00                    510.5      99.960       0.02
      440.5     100.038       0.00                    515.1      99.951       0.03
      445.5     100.038       0.00                    520.5      99.942       0.02
      450.3     100.034       0.01                    525.1      99.935       0.01
      455.1     100.033       0.02                    530.5      99.928       0.03
      460.8     100.029       0.01                    535.7      99.920       0.02
      465.6     100.020       0.02                    540.2      99.915       0.02
      470.4     100.018       0.02                    545.6      99.910       0.02
                                                      550.2      99.901       0.03
```

```
Sample: RUN#54                                    Date: 10-Oct-85   Time: 10:09:52
Size: 55.31 mg                                    File: COKE.02 T.LYNCH.08
Rate: 15C/MIN                                     Operator: JT
Program: General Analysis V1.0                    Plotted:  1-Nov-85   15:08:51

      Temp      Sig-A     Deriv-A                    Temp      Sig-A     Deriv-A
     [deg]       [%]      [%/min]                   [deg]       [%]      [%/min]

     550.2     99.901       0.03                    625.3     98.266       1.29
     555.6     99.893       0.03                    630.2     97.644       0.34
     560.1     99.882       0.03                    635.3     97.536       0.07
     565.3     99.877       0.02                    640.2     97.512       0.03
     570.4     99.866       0.02                    645.3     97.501       0.02
     575.1     99.861       0.03                    650.1     97.496       0.02
     580.3     99.852       0.01                    655.3     97.494       0.02
     585.6     99.848       0.01                    660.4     97.483       0.00
     590.4     99.845       0.04                    665.1     97.480       0.01
     595.2     99.826       0.03                    670.3     97.471       0.01
     600.1     99.816       0.03                    675.1     97.467       0.01
     605.3     99.779       0.15                    680.1     97.462       0.01
     610.4     99.658       0.43                    685.2     97.458       0.01
     615.3     99.400       0.92                    690.3     97.449       0.01
     620.5     98.915       1.42                    695.4     97.444       0.01
                                                    700.1     97.435       0.01
```

Sample: RUN#60
Size: 57.81 mg
Rate: 15C/MIN
Program: TGA Analysis V2.0

TGA

Date: 10-Oct-85 Time: 15:59:12
File: COKE.04 T.LYNCH.08
Operator: JT
Plotted: 11-Oct-85 7:51:34



DuPont 1090

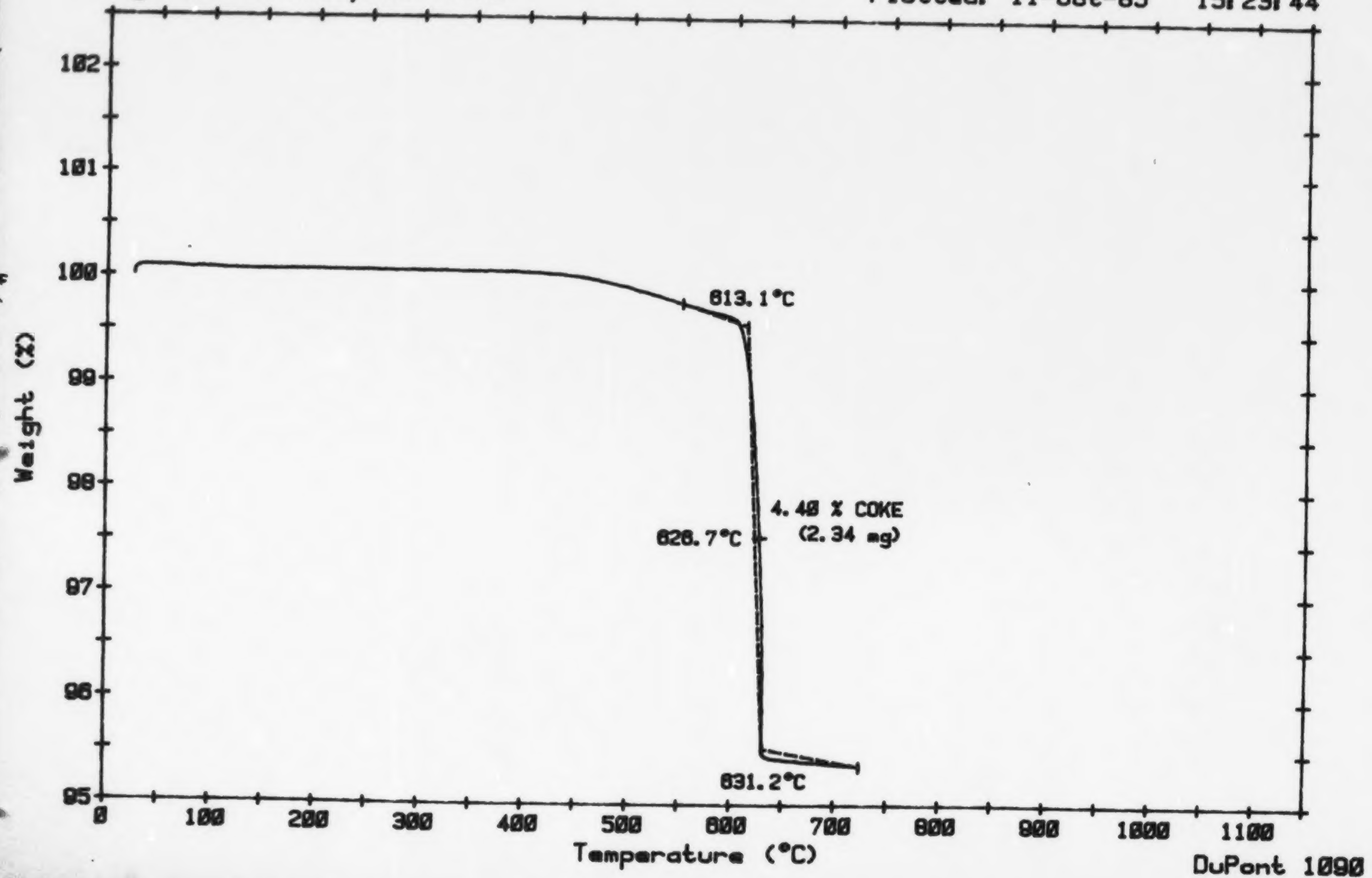
```
Sample: RUN#60                                  Date: 10-Oct-85   Time: 15:59:12
Size: 57.81 mg                                  File: COKE.04 T.LYNCH.08
Rate: 15C/MIN                                   Operator: JT
Program: General Analysis V1.0                  Plotted: 1-Nov-85   15:52:36

     Temp      Sig-A     Deriv-A                  Temp      Sig-A     Deriv-A
     [deg]      [%]      [%/min]                  [deg]      [%]      [%/min]

     400.0    100.102     0.00                    475.1    100.099     0.01
     405.2    100.104     0.00                    480.2    100.095     0.01
     410.3    100.104     0.00                    485.3    100.090     0.01
     415.4    100.102     0.01                    490.4    100.090     0.00
     420.5    100.102    -0.01                    495.4    100.088     0.01
     425.6    100.104     0.00                    500.4    100.083     0.01
     430.7    100.106     0.00                    505.4    100.081     0.02
     435.7    100.102     0.01                    510.4    100.076     0.00
     440.0    100.102    -0.01                    515.3    100.078     0.01
     445.2    100.102     0.00                    520.3    100.073     0.02
     450.3    100.102     0.00                    525.3    100.067     0.01
     455.3    100.100     0.00                    530.2    100.066     0.00
     460.5    100.100     0.01                    535.1    100.064     0.01
     465.6    100.097     0.00                    540.1    100.057     0.02
     470.8    100.099     0.00                    545.0    100.054     0.02
                                                  550.7    100.048     0.01
```

```
Sample: RUN#60                                  Date: 10-Oct-85    Time: 15:59:12
Size: 57.81 mg                                  File: COKE.04 T.LYNCH.08
Rate: 15C/MIN                                   Operator: JT
Program: General Analysis V1.0                  Plotted:  1-Nov-85    16:03:26

     Temp       Sig-A      Deriv-A                    Temp       Sig-A      Deriv-A
     [deg]       [%]       [%/min]                    [deg]       [%]       [%/min]

     550.7     100.048      0.01                      625.1      99.183      0.10
     555.5     100.043      0.01                      630.3      99.154      0.04
     560.4     100.038      0.02                      635.5      99.140      0.02
     565.3     100.035      0.01                      640.1      99.133      0.02
     570.2     100.029      0.00                      645.3      99.130      0.01
     575.0     100.028      0.01                      650.0      99.125      0.01
     580.7     100.024      0.01                      655.3      99.118      0.01
     585.6     100.019      0.02                      660.4      99.116      0.01
     590.2     100.016      0.02                      665.5      99.111      0.01
     595.3     100.005      0.02                      670.2      99.109      0.00
     600.3      99.995      0.02                      675.0      99.106      0.01
     605.1      99.972      0.10                      680.2      99.102      0.00
     610.6      99.886      0.30                      685.2      99.100      0.01
     615.3      99.739      0.59                      690.4      99.092      0.01
     620.5      99.436      0.78                      695.1      99.087      0.01
                                                      700.2      99.083      0.00
```

Sample: RUN#72
Size: 53.19 mg
Rate: 15C/MIN
Program: TGA Analysis V2.0

TGA

Date: 11-Oct-85 Time: 14:34:14
File: COKE.09 T.LYNCH.08
Operator: JT
Plotted: 11-Oct-85 15:23:44



DuPont 1090

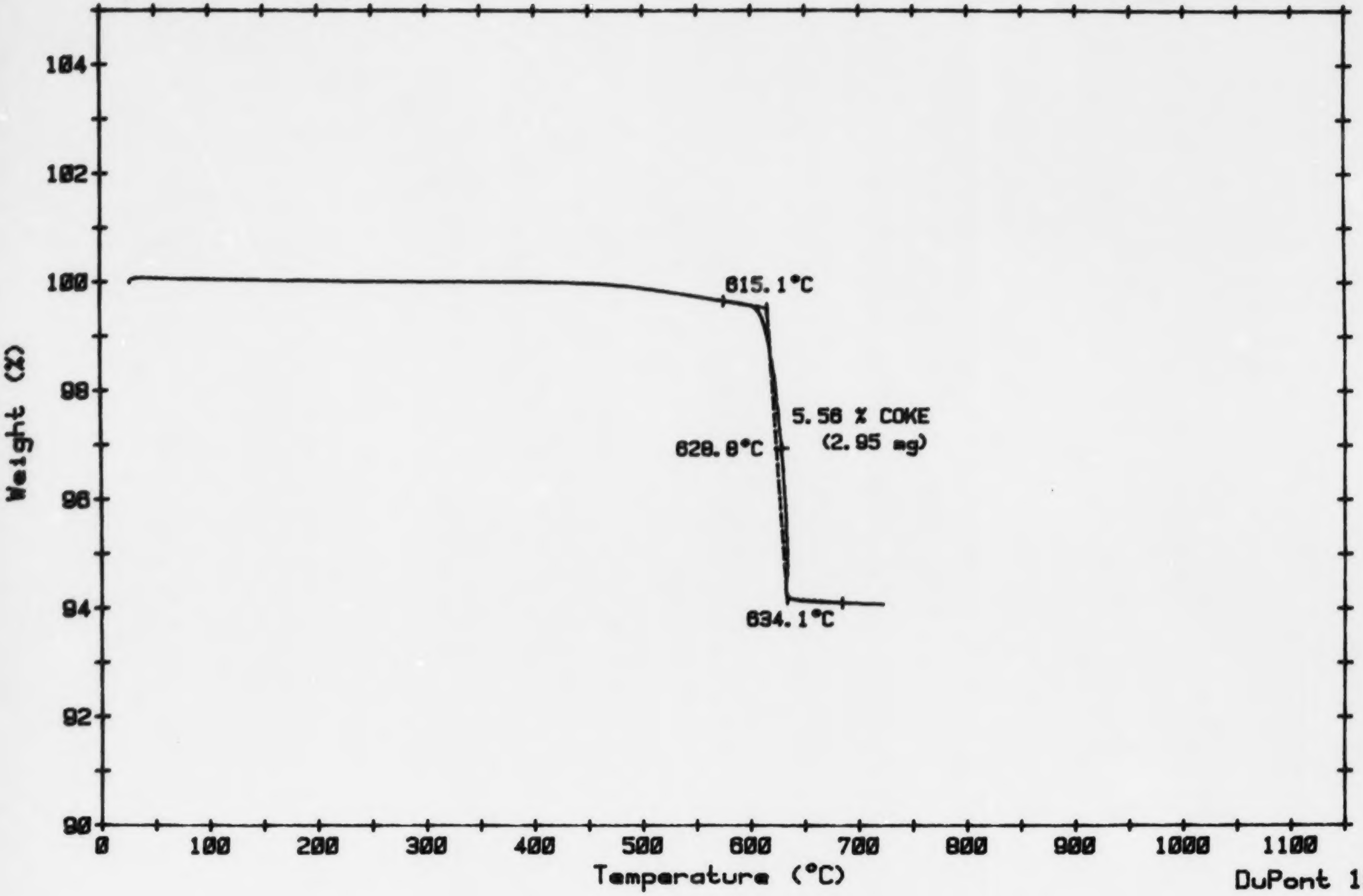
```
Sample: RUN#72                                   Date: 11-Oct-85    Time: 14:34:14
Size: 53.19 mg                                   File: COKE.09 T.LYNCH.08
Rate: 15C/MIN                                    Operator: JT
Program: General Analysis V1.0                   Plotted:  4-Nov-85     9:50:51

      Temp      Sig-A     Deriv-A                     Temp      Sig-A     Deriv-A
      [deg]      [%]      [%/min]                     [deg]      [%]      [%/min]

      400.3    100.055      0.00                      475.3     99.972      0.04
      405.5    100.051      0.01                      480.3     99.959      0.04
      410.6    100.051      0.00                      485.4     99.947      0.04
      415.8    100.049      0.01                      490.5     99.932      0.03
      420.8    100.045      0.02                      495.6     99.925      0.04
      425.3    100.041      0.01                      500.7     99.910      0.05
      430.4    100.038      0.02                      505.7     99.893      0.05
      435.5    100.032      0.02                      510.8     99.887      0.02
      440.5    100.026      0.01                      515.8     99.874      0.06
      445.6    100.023      0.02                      520.7     99.855      0.05
      450.7    100.015      0.03                      525.8     99.842      0.04
      455.0    100.008      0.03                      530.8     99.829      0.06
      460.1    100.000      0.03                      535.7     99.812      0.05
      465.2     99.992      0.03                      540.6     99.797      0.05
      470.3     99.979      0.04                      545.5     99.784      0.05
                                                      550.5     99.771      0.05
```

```
Sample: RUN#72                                  Date: 11-Oct-85    Time: 14:34:14
Size: 53.19 mg                                  File: COKE.09 T.LYNCH.08
Rate: 15C/MIN                                   Operator: JT
Program: General Analysis V1.0                  Plotted: 4-Nov-85    10:13:49

     Temp      Sig-A      Deriv-A                   Temp      Sig-A      Deriv-A
     [deg]      [%]       [%/min]                   [deg]      [%]       [%/min]

     550.5     99.771       0.05                    625.1     97.836       2.90
     555.4     99.754       0.05                    630.0     96.703       2.50
     560.2     99.741       0.04                    635.3     95.456       0.04
     565.1     99.727       0.05                    640.4     95.443       0.02
     570.0     99.714       0.04                    645.1     95.435       0.01
     575.6     99.703       0.04                    650.3     95.430       0.02
     580.6     99.690       0.04                    655.4     95.426       0.01
     585.4     99.675       0.03                    660.0     95.424      -0.01
     590.2     99.660       0.03                    665.2     95.419       0.02
     595.3     99.652       0.05                    670.4     95.411       0.01
     600.2     99.632       0.05                    675.1     95.409       0.01
     605.6     99.558       0.35                    680.3     95.405       0.01
     610.2     99.395       0.84                    685.4     95.404       0.01
     615.6     99.030       1.65                    690.0     95.400       0.01
     620.4     98.522       2.42                    695.2     95.392       0.02
                                                    700.4     95.388       0.01
```

Sample: RUN#80
Size: 53.03 mg
Rate: 15C/MIN
Program: TGA Analysis V2.0

TGA

Date: 11-Oct-85 Time: 13:12:48
File: COKE.08 T.LYNCH.08
Operator: JT
Plotted: 11-Oct-85 14:11:03



DuPont 1090

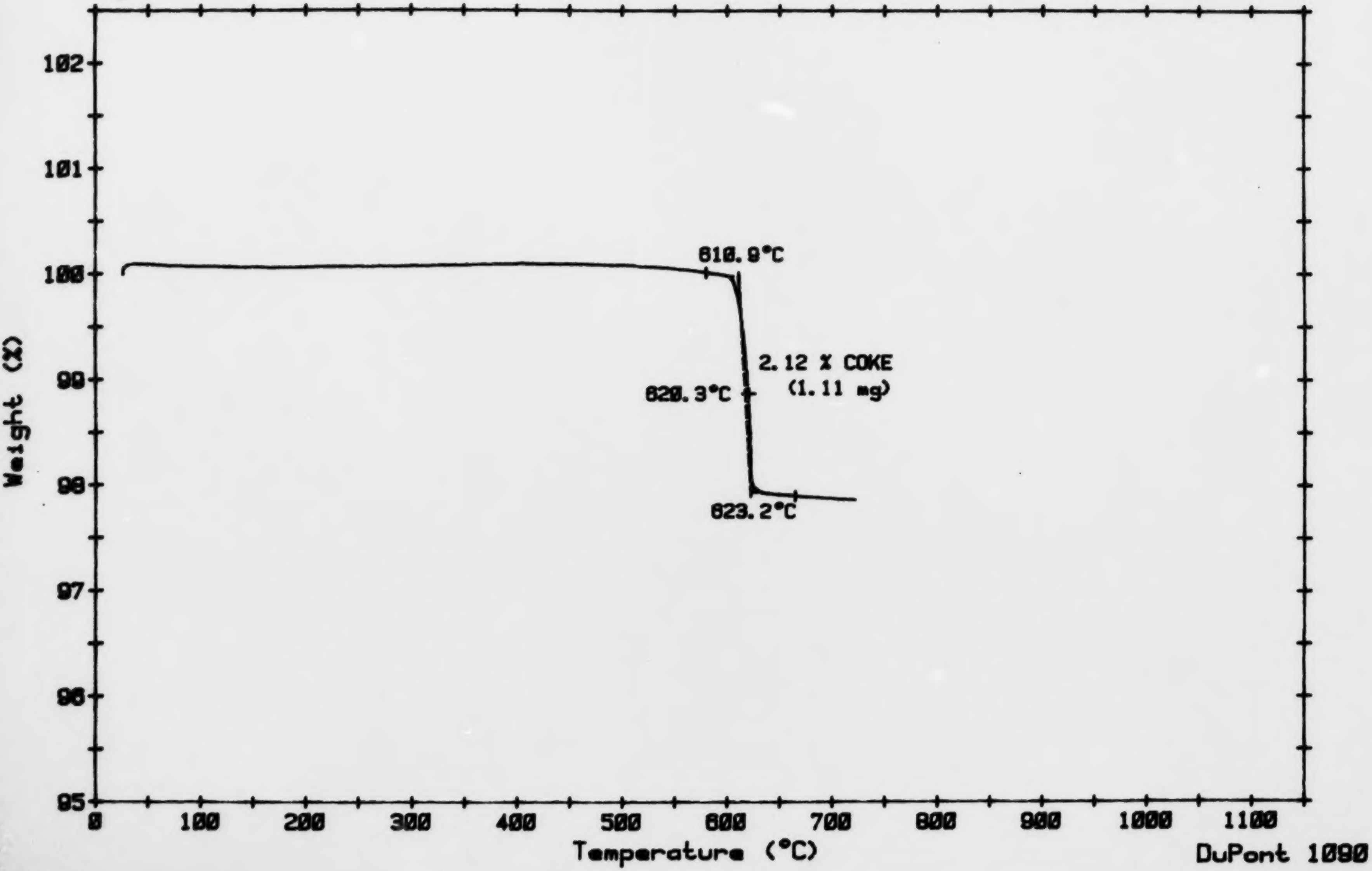
```
Sample: RUN#80                                      Date: 11-Oct-85    Time: 13:12:48
Size: 53.03 mg                                      File: COKE.08 T.LYNCH.08
Rate: 15C/MIN                                       Operator: JT
Program: General Analysis V1.0                      Plotted: 5-Nov-85     7:35:28

      Temp        Sig-A       Deriv-A                    Temp        Sig-A       Deriv-A
     [deg]         [%]       [%/min]                    [deg]         [%]       [%/min]

     400.2      100.009        0.01                     475.4       99.947        0.03
     405.4      100.008        0.01                     480.4       99.936        0.04
     410.6      100.006        0.00                     485.4       99.928        0.02
     415.9      100.004        0.01                     490.5       99.915        0.04
     420.2      100.002        0.01                     495.6       99.900        0.05
     425.4      100.002        0.01                     500.6       99.887        0.05
     430.4       99.998        0.01                     505.5       99.876        0.05
     435.5       99.996        0.01                     510.4       99.859        0.05
     440.7       99.991        0.01                     515.3       99.847        0.04
     445.8       99.985        0.02                     520.4       99.834        0.06
     450.0       99.985        0.01                     525.4       99.819        0.05
     455.1       99.979        0.02                     530.3       99.802        0.06
     460.2       99.972        0.02                     535.2       99.787        0.06
     465.3       99.964        0.03                     540.1       99.768        0.06
     470.3       99.955        0.03                     545.0       99.753        0.04
                                                        550.8       99.734        0.06
```

```
Sample: RUN#80                                        Date: 11-Oct-85    Time: 13:12:48
Size: 53.03 mg                                        File: COKE.08 T.LYNCH.08
Rate: 15C/MIN                                         Operator: JT
Program: General Analysis V1.0                        Plotted: 5-Nov-85     8:20:20

     Temp      Sig-A     Deriv-A                         Temp      Sig-A     Deriv-A
     [deg]      [%]      [%/min]                         [deg]      [%]      [%/min]

     550.8     99.734      0.06                          625.4     97.651      3.36
     555.7     99.717      0.05                          630.2     96.581      3.54
     560.6     99.700      0.05                          635.2     94.202      0.16
     565.6     99.685      0.05                          640.1     94.153      0.05
     570.4     99.670      0.05                          645.3     94.136      0.03
     575.1     99.657      0.05                          650.0     94.128      0.01
     580.0     99.642      0.04                          655.1     94.121      0.01
     585.6     99.627      0.05                          660.3     94.113      0.00
     590.4     99.610      0.04                          665.3     94.109      0.02
     595.0     99.595      0.07                          670.1     94.106      0.01
     600.0     99.570      0.04                          675.4     94.100      0.02
     605.0     99.517      0.20                          680.0     94.098      0.01
     610.4     99.338      0.86                          685.0     94.094      0.01
     615.8     98.981      1.73                          690.1     94.090      0.00
     620.7     98.416      2.64                          695.3     94.085      0.00
                                                         700.4     94.083      0.01
```

Sample: RUN#79
Size: 52.24 mg
Rate: 15C/MIN
Program: TGA Analysis V2.0

TGA

Date: 11-Oct-85 Time: 11:26:39
File: COKE.07 T.LYNCH.08
Operator: JT
Plotted: 11-Oct-85 12:57:03



DuPont 1090

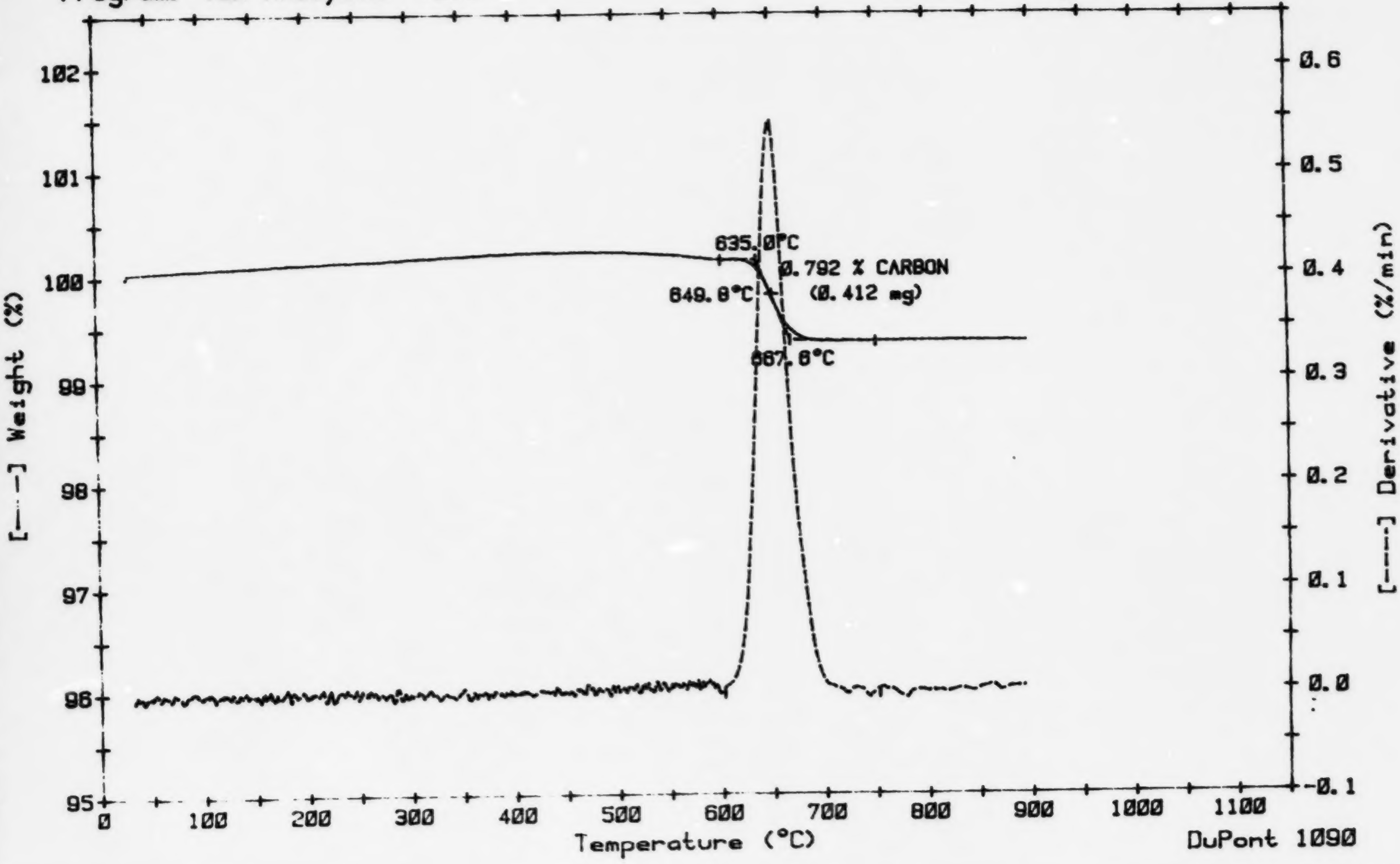
```
Sample: RUN#79                                        Date: 11-Oct-85    Time: 11:26:39
Size: 52.24 mg                                        File: COKE.07 T.LYNCH.08
Rate: 15C/MIN                                         Operator: JT
Program: General Analysis V1.0                        Plotted: 5-Nov-85    8:45:47

                                                      Temp     Sig-A     Deriv-A
     Temp     Sig-A      Deriv-A                      [deg]     [%]      [%/min]
     [deg]     [%]       [%/min]
                                                      475.1   100.086     0.00
     400.6   100.100     -0.02                        480.0   100.086     0.00
     405.8   100.100      0.00                        485.0   100.084     0.01
     410.1   100.100      0.00                        490.0   100.084     0.00
     415.3   100.096      0.01                        495.1   100.086     0.01
     420.5   100.096      0.00                        500.2   100.082     0.00
     425.6   100.092      0.01                        505.2   100.080     0.00
     430.8   100.096     -0.01                        510.4   100.078     0.02
     435.0   100.096      0.01                        515.4   100.075     0.01
     440.2   100.092      0.00                        520.4   100.071     0.01
     445.4   100.094      0.00                        525.5   100.068     0.01
     450.5   100.092      0.00                        530.6   100.065     0.00
     455.6   100.094      0.00                        535.6   100.061     0.01
     460.8   100.092      0.01                        540.5   100.061     0.01
     465.0   100.092      0.00                        545.5   100.058     0.02
     470.1   100.090      0.02                        550.5   100.052     0.01
```

```
Sample: RUN#79                                    Date: 11-Oct-85    Time: 11:26:39
Size: 52.24 mg                                    File: COKE.07 T.LYNCH.08
Rate: 15C/MIN                                     Operator: JT
Program: General Analysis V1.0                    Plotted:  5-Nov-85    10:30:46

     Temp      Sig-A      Deriv-A                      Temp      Sig-A      Deriv-A
     [deg]      [%]       [%/min]                      [deg]      [%]       [%/min]

     550.5    100.052       0.01                       625.1     97.992       0.16
     555.5    100.046       0.02                       630.0     97.940       0.05
     560.4    100.042       0.02                       635.1     97.921       0.04
     565.4    100.036       0.02                       640.2     97.911       0.01
     570.3    100.029       0.02                       645.0     97.908       0.01
     575.8    100.023       0.02                       650.1     97.904       0.01
     580.1    100.017       0.01                       655.3     97.902       0.01
     585.1    100.011       0.03                       660.1     97.898       0.01
     590.0    100.004       0.02                       665.3     97.892       0.00
     595.5     99.996       0.04                       670.0     97.887       0.01
     600.1     99.983       0.02                       675.3     97.883       0.01
     605.2     99.941       0.23                       680.5     97.879       0.01
     610.5     99.755       0.79                       685.1     97.877       0.00
     615.5     99.416       1.50                       690.3     97.875       0.00
     620.3     98.869       1.95                       695.4     97.873       0.01
                                                       700.1     97.867       0.00
```

Sample: RUN#43
Size: 52.01 mg
Rate: 05C/MIN
Program: TGA Analysis V2.0

TGA

Date: 27-Aug-85 Time: 11:04:17
File: PEL-COKE.16 TLYNCH.06
Operator: JT
Plotted: 27-Aug-85 13:16:21



DuPont 1090

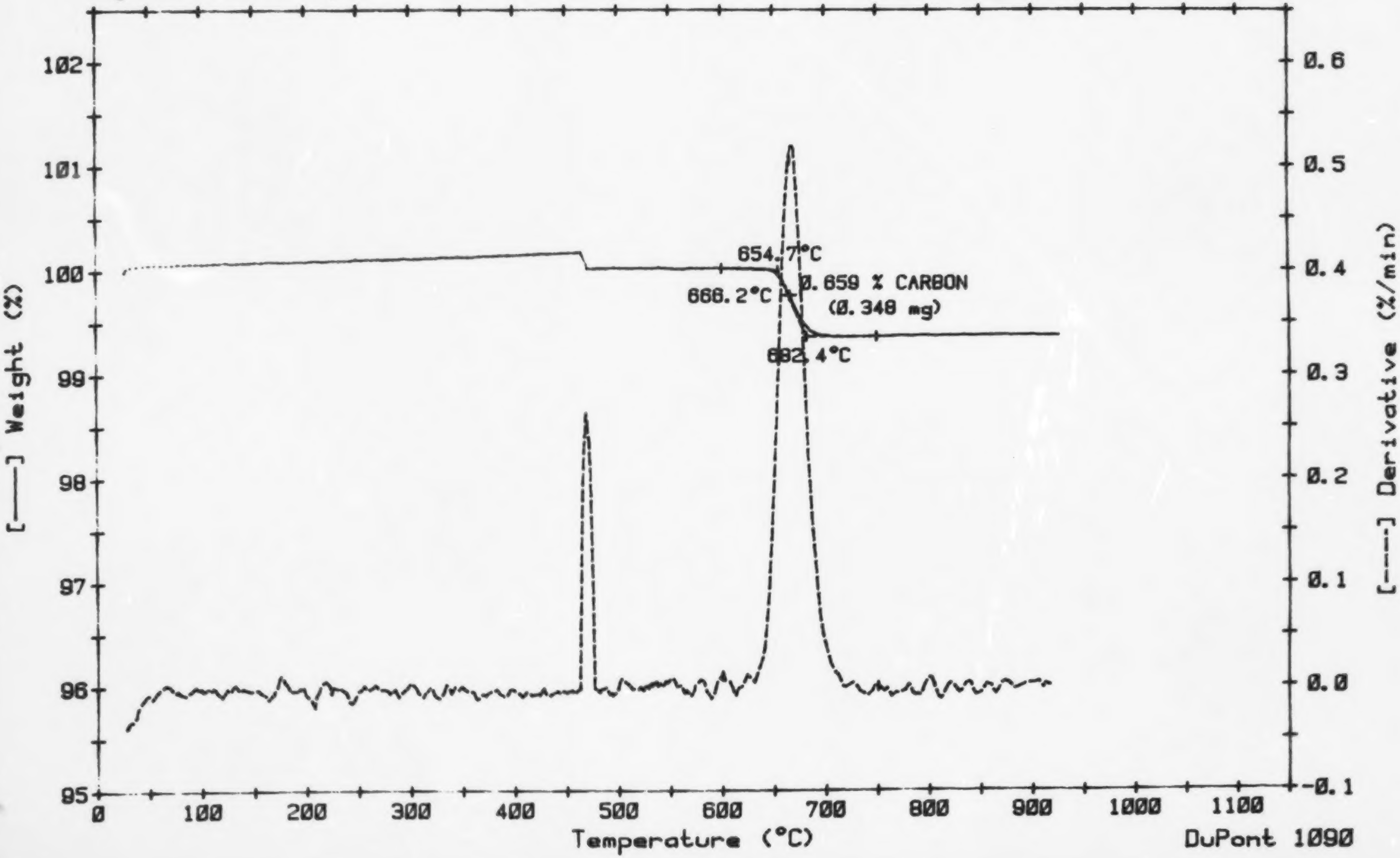
```
Sample: RUN#43                                   Date: 27-Aug-85    Time: 11:04:17
Size: 52.01 mg                                   File: PEL-COKE.16 TLYNCH.06
Rate: 05C/MIN                                    Operator: JT
Program: General Analysis V1.0                  Plotted: 3-Sep-85   13:45:04

    Temp      Sig-A      Deriv-A                 Temp      Sig-A      Deriv-A
    [deg]     [%]        [%/min]                 [deg]     [%]        [%/min]

    400.2     100.213    0.00                    500.1     100.219    0.00
    405.1     100.217    0.00                    505.2     100.215    0.00
    410.2     100.217    0.00                    510.1     100.215    0.00
    415.1     100.217    0.00                    515.0     100.213    0.00
    420.0     100.221    0.00                    520.0     100.210    0.00
    425.1     100.223    0.00                    525.0     100.204    0.00
    430.1     100.221    0.00                    530.0     100.204    0.00
    435.0     100.221    0.00                    535.2     100.202    0.01
    440.1     100.221    0.00                    540.2     100.196    0.00
    445.2     100.223    0.00                    545.0     100.194    0.00
    450.0     100.225    0.00                    550.1     100.192    0.01
    455.1     100.225    0.00                    555.0     100.186    0.01
    460.1     100.227    0.00                    560.1     100.183    0.00
    465.2     100.225    0.00                    565.2     100.179    0.00
    470.2     100.225    0.00                    570.1     100.173    0.00
    475.1     100.221    0.00                    575.1     100.165    0.00
    480.2     100.225    0.00                    580.0     100.163    0.01
    485.2     100.221    0.00                    585.1     100.156    0.00
    490.2     100.219    0.00                    590.1     100.148    0.01
    495.2     100.219    0.00                    595.1     100.142    0.01
                                                 600.7     100.146    0.00
```

```
Sample: RUN#43                                        Date: 27-Aug-85    Time: 11:04:17
Size: 52.01 mg                                        File: PEL-COKE.16 TLYNCH.CS
Rate: 05C/MIN                                         Operator: JT
Program: General Analysis V1.0                        Plotted:  3-Sep-85   13:52:08

     Temp      Sig-A      Deriv-A                         Temp      Sig-A      Deriv-A
     [deg]      [%]       [%/min]                         [deg]      [%]       [%/min]

     600.7    100.146       0.00                          675.9     99.416       0.08
     605.9    100.144       0.00                          680.4     99.389       0.06
     610.7    100.142       0.00                          685.0     99.368       0.04
     615.8    100.142       0.01                          690.8     99.358       0.01
     621.2    100.136       0.02                          695.5     99.356       0.01
     625.3    100.129       0.04                          700.2     99.350       0.00
     630.8    100.108       0.08                          706.1     99.350       0.00
     636.3    100.060       0.15                          710.8     99.352       0.00
     640.3    100.006       0.21                          715.5     99.350       0.00
     645.8     99.906       0.28                          720.2     99.352       0.00
     651.1     99.785       0.29                          726.1     99.352       0.00
     656.0     99.675       0.25                          730.7     99.352       0.00
     660.7     99.583       0.20                          735.3     99.350       0.00
     665.4     99.514       0.15                          740.0     99.352       0.00
     670.0     99.462       0.11                          745.7     99.354       0.00
                                                          750.3     99.354       0.00
```

Sample: RUN#43
Size: 52.80 mg
Rate: 15C/MIN
Program: TGA Analysis V2.0

TGA

Date: 3-Sep-85 Time: 12:07:17
File: PEL-COKE.17 TLYNCH.06
Operator: JT
Plotted: 3-Sep-85 13:19:29



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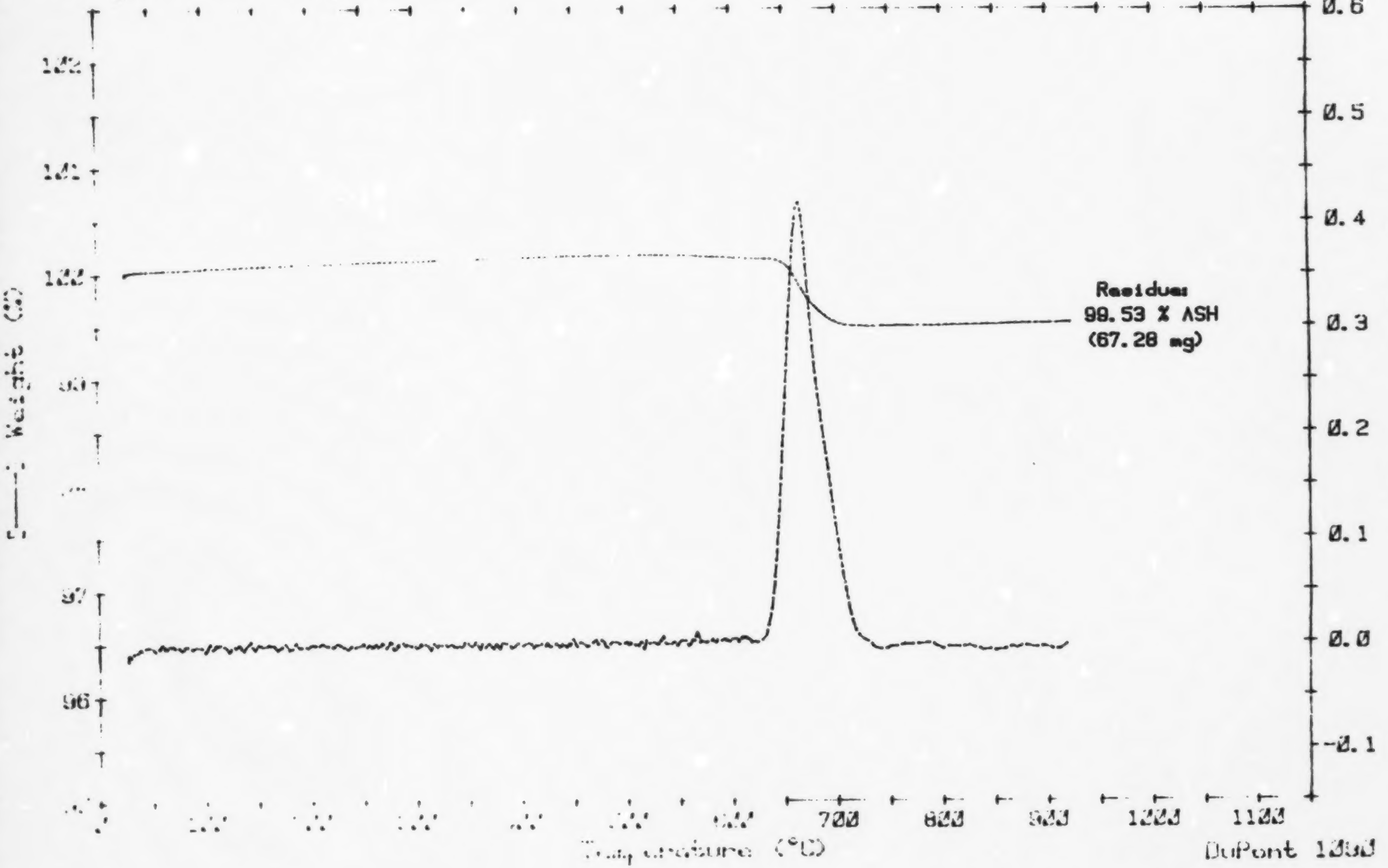
```
Sample: RUN#43                                      Date:   3-Sep-85    Time: 12:07:17
Size: 52.80 mg                                      File: PEL-COKE.17 TLYNCH.06
Rate: 15C/MIN                                       Operator: JT
Program: General Analysis V1.0                      Plotted:   3-Sep-85    14:22:09

     Temp      Sig-A      Deriv-A                    Temp      Sig-A      Deriv-A
     [deg]      [%]       [%/min]                    [deg]      [%]       [%/min]

     400.0    100.146       0.00                     500.6    100.025       0.00
     405.7    100.148      -0.01                     505.2    100.023       0.00
     410.5    100.152      -0.01                     510.7    100.017       0.00
     415.4    100.150       0.00                     515.3    100.021       0.00
     420.3    100.153      -0.01                     520.7    100.023       0.00
     425.2    100.157       0.00                     525.4    100.023       0.00
     430.0    100.161       0.00                     530.1    100.025       0.00
     435.6    100.165      -0.01                     535.4    100.019       0.00
     440.4    100.165       0.00                     540.0    100.021       0.00
     445.2    100.165      -0.01                     545.4    100.021       0.00
     450.1    100.169       0.00                     550.7    100.019       0.01
     455.6    100.170       0.00                     555.3    100.017       0.00
     460.5    100.170       0.00                     560.6    100.017       0.00
     465.2    100.170      -0.01                     565.1    100.015       0.00
     470.7    100.015       0.24                     570.5    100.017       0.00
     475.5    100.015       0.00                     575.0    100.023       0.00
     480.2    100.015       0.00                     580.1    100.017       0.01
     485.6    100.015       0.00                     585.4    100.019       0.00
     490.4    100.021       0.00                     590.7    100.023      -0.01
     495.1    100.019      -0.01                     595.2    100.028       0.00
                                                     600.4    100.021       0.00
```

```
Sample: RUN#43                                        Date:   3-Sep-85    Time: 12:07:17
Size:  52.80 mg                                       File:  PEL-COKE.17 TLYNCH.06
Rate:  15C/MIN                                        Operator:  JT
Program:  General Analysis V1.0                       Plotted:   3-Sep-85    14:29:01

     Temp       Sig-A      Deriv-A                         Temp       Sig-A      Deriv-A
     [deg]       [%]       [%/min]                         [deg]       [%]       [%/min]

     600.4     100.021       0.00                          675.8      99.544       0.21
     605.5     100.017       0.00                          680.4      99.479       0.14
     610.7     100.017       0.00                          686.1      99.415       0.09
     615.1     100.017      -0.02                          690.8      99.380       0.05
     620.5     100.019       0.02                          695.4      99.373       0.03
     625.0     100.017       0.00                          700.1      99.364       0.01
     630.9     100.015       0.01                          705.9      99.360       0.01
     635.9     100.013       0.00                          710.6      99.354       0.00
     641.0     100.013       0.01                          715.3      99.356       0.00
     646.2     100.004       0.04                          720.0      99.358       0.00
     650.2      99.991       0.08                          725.9      99.358       0.00
     655.5      99.938       0.15                          730.6      99.356       0.01
     660.8      99.869       0.23                          735.2      99.354      -0.01
     666.2      99.758       0.29                          741.1      99.362       0.00
     671.1      99.640       0.26                          745.8      99.360       0.00
                                                           750.5      99.362       0.00
```

Sample: RUN#54
Size: 67.60 mg
Rate: 05C/MIN
Program: TGA Analysis V[illegible]

TGA

Date: 28-Aug-85 Time: 8:42:16
File: PEL-COKE.30 TLYNC



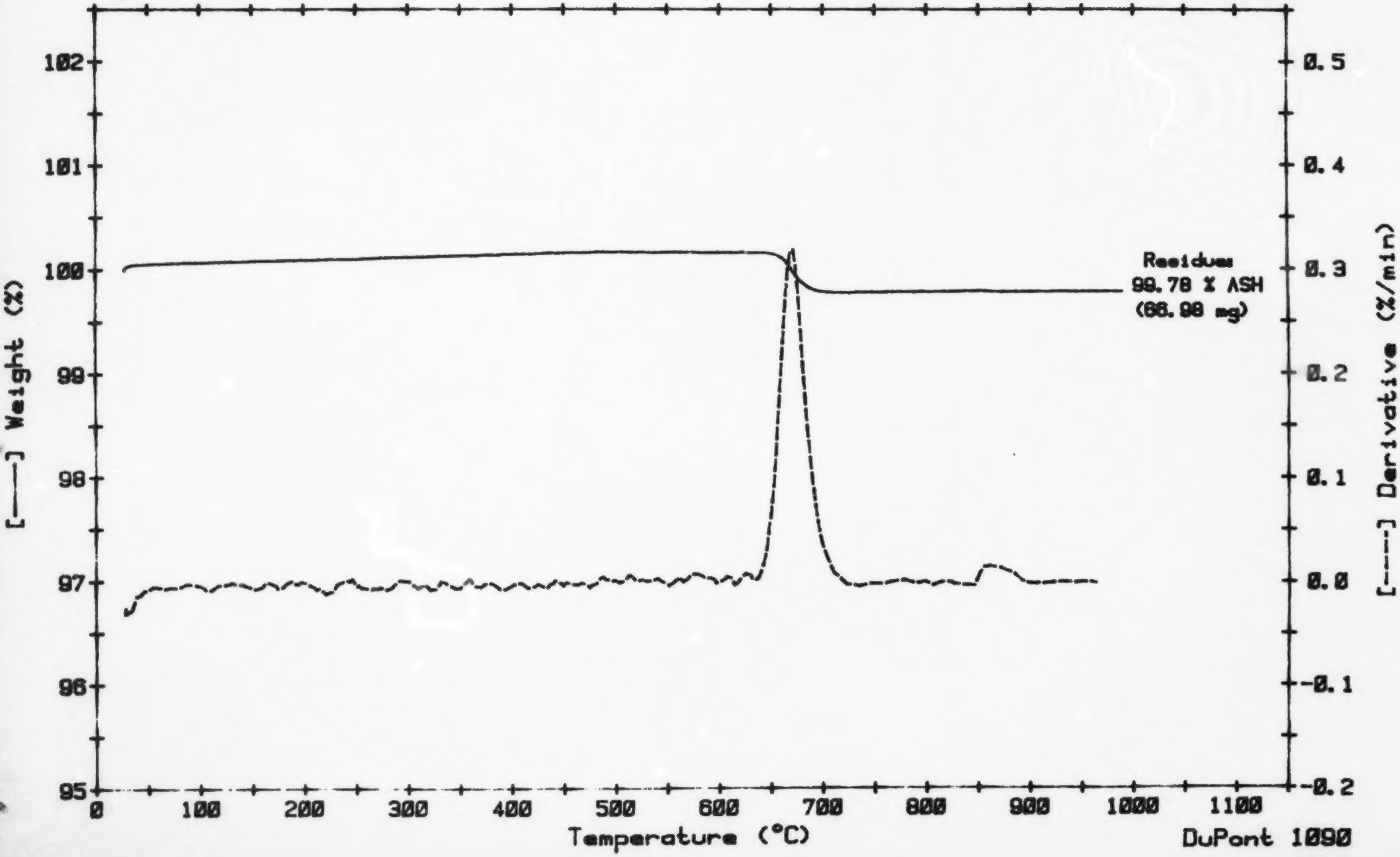
```
Sample: RUN#34                                       Date: 28-Aug-85    Time:  8:42:16
Size: 67.60 mg                                       File: PEL-COKE.30 TLYNCH.06
Rate: 05C/MIN                                        Operator: JT
Program: General Analysis V1.0                       Plotted:  3-Sep-85    15:37:32

     Temp       Sig-A      Deriv-A                        Temp       Sig-A      Deriv-A
     [deg]       [%]       [%/min]                        [deg]       [%]       [%/min]

     400.1     100.155       0.00                         500.1     100.175       0.00
     405.2     100.157       0.00                         505.1     100.175       0.00
     410.0     100.157       0.00                         510.1     100.176       0.00
     415.1     100.161       0.00                         515.2     100.178      -0.01
     420.2     100.161       0.00                         520.0     100.178       0.00
     425.1     100.164       0.00                         525.1     100.178       0.00
     430.2     100.166       0.00                         530.1     100.178       0.00
     435.0     100.167       0.00                         535.1     100.175       0.00
     440.0     100.169       0.00                         540.0     100.175       0.00
     445.1     100.172       0.00                         545.2     100.173       0.00
     450.1     100.170       0.00                         550.1     100.175       0.00
     455.1     100.172       0.00                         555.0     100.172       0.00
     460.3     100.172       0.00                         560.1     100.173       0.00
     465.2     100.173       0.00                         565.0     100.170       0.00
     470.2     100.172       0.00                         570.0     100.163       0.00
     475.1     100.172       0.00                         575.1     100.163       0.00
     480.1     100.172       0.00                         580.0     100.161       0.00
     485.1     100.173       0.00                         585.2     100.158       0.00
     490.1     100.173       0.00                         590.0     100.155       0.00
     495.2     100.175       0.00                         595.1     100.155       0.00
                                                          600.0     100.151       0.00
```

```
Sample: RUN#34                                       Date: 28-Aug-85   Time:  8:42:16
Size: 67.60 mg                                       File: PEL-COKE.30 TLYNCH.06
Rate: 05C/MIN                                        Operator: JT
Program: General Analysis V1.0                       Plotted:  3-Sep-85   15:44:35

      Temp        Sig-A       Deriv-A                    Temp        Sig-A       Deriv-A
      [deg]        [%]        [%/min]                    [deg]        [%]        [%/min]

      600.0      100.151         0.00                    676.0       99.728         0.15
      605.2      100.149         0.00                    680.8       99.676         0.12
      610.0      100.145         0.01                    685.6       99.632         0.10
      615.1      100.139         0.00                    690.3       99.593         0.09
      620.5      100.142         0.00                    695.1       99.561         0.07
      625.3      100.139         0.00                    700.9       99.531         0.05
      631.0      100.138         0.00                    705.6       99.516         0.03
      636.1      100.139         0.01                    710.3       99.503         0.02
      640.1      100.133         0.02                    715.0       99.501         0.01
      645.8      100.120         0.05                    720.8       99.499         0.00
      651.2      100.090         0.11                    725.5       99.499         0.00
      655.5      100.050         0.16                    730.1       99.497         0.00
      661.1       99.987         0.23                    735.9       99.497         0.00
      665.0       99.895         0.23                    740.7       99.500        -0.01
      670.0       99.812         0.19                    745.3       99.500         0.00
                                                         751.1       99.501         0.00
```

Sample: RUN#17
Size: 67.13 mg
Rate: 15C/MIN
Program: TGA Analysis V2.0

TGA

Date: 28-Aug-85 Time: 13:54:44
File: PEL-COKE.40 TLYNCH.06
Operator: JT
Plotted: 28-Aug-85 14:46:49



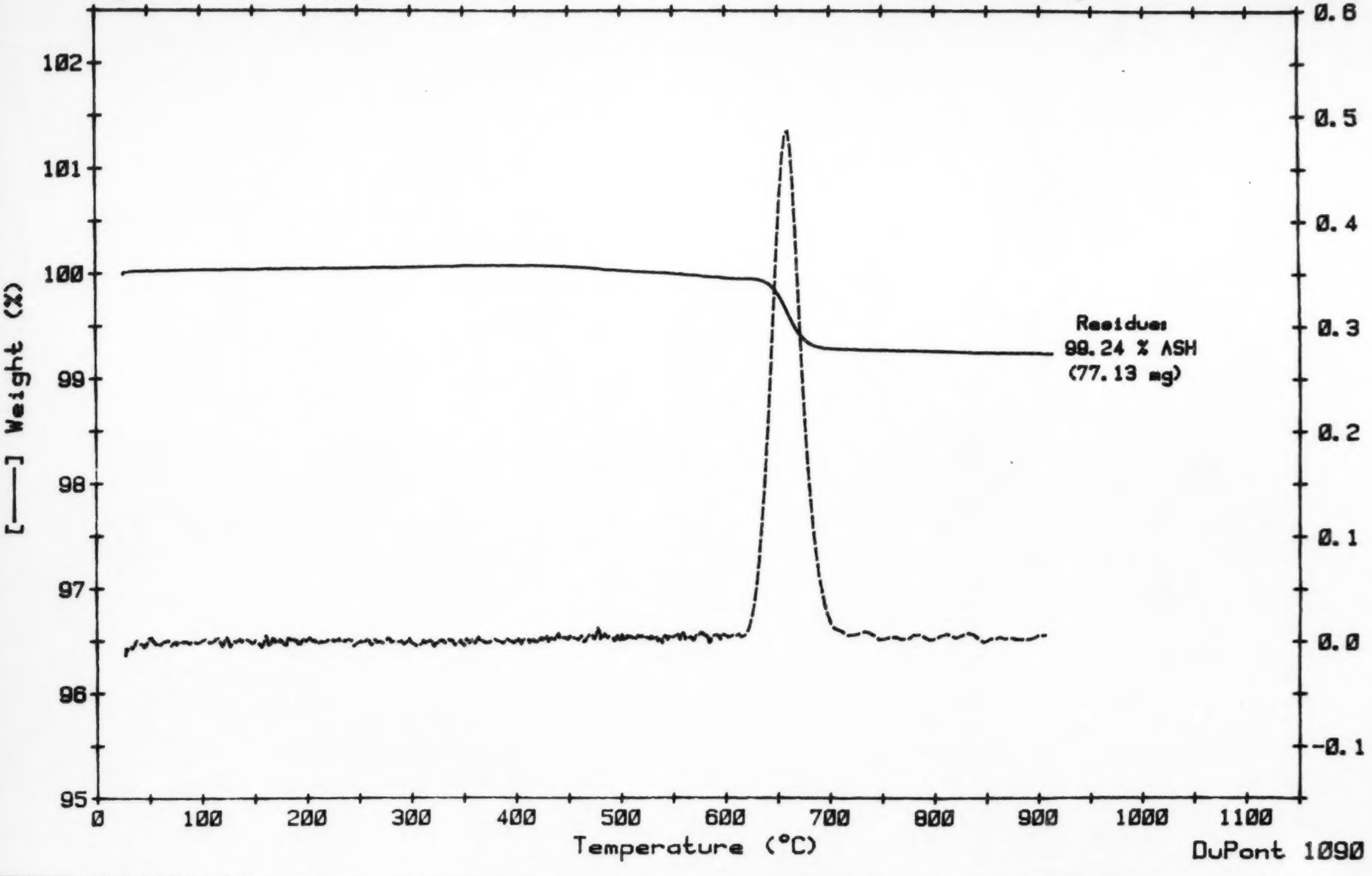
```
Sample: RUN#17                                    Date: 28-Aug-85    Time: 13:54:44
Size: 67.13 mg                                    File: PEL-COKE.40 TLYNCH.06
Rate: 15C/MIN                                     Operator: JT
Program: General Analysis V1.0                    Plotted:  4-Sep-85     8:31:23

      Temp       Sig-A       Deriv-A                    Temp       Sig-A       Deriv-A
      [deg]       [%]        [%/min]                    [deg]       [%]        [%/min]

      400.7     100.146        0.00                     500.2     100.164        0.00
      405.8     100.146        0.00                     505.7     100.165        0.00
      410.7     100.147        0.00                     510.4     100.164        0.00
      415.7     100.149        0.00                     515.1     100.162        0.00
      420.6     100.152        0.00                     520.5     100.162        0.00
      425.6     100.152        0.00                     525.1     100.164        0.00
      430.5     100.153        0.00                     530.5     100.162        0.00
      435.4     100.156        0.00                     535.1     100.164        0.00
      440.3     100.158        0.00                     540.5     100.162        0.00
      445.1     100.156        0.00                     545.1     100.162        0.00
      450.0     100.156        0.00                     550.4     100.164        0.00
      455.6     100.161        0.00                     555.0     100.165        0.00
      460.4     100.159        0.00                     560.4     100.165        0.00
      465.2     100.162        0.00                     565.7     100.165        0.00
      470.0     100.161        0.00                     570.3     100.165        0.00
      475.6     100.162        0.00                     575.6     100.162        0.00
      480.3     100.165        0.00                     580.0     100.161        0.00
      485.1     100.167        0.00                     585.4     100.159        0.00
      490.7     100.162        0.00                     590.7     100.159        0.00
      495.5     100.165        0.00                     595.2     100.158        0.00
                                                        600.4     100.159        0.00
```

```
Sample: RUN#17                                        Date: 28-Aug-85    Time: 13:54:44
Size: 67.13 mg                                        File: PEL-COKE.40 TLYNCH.06
Rate: 15C/MIN                                         Operator: JT
Program: General Analysis V1.0                        Plotted:   4-Sep-85     8:38:20

      Temp      Sig-A      Deriv-A                      Temp      Sig-A     Deriv-A
      [deg]      [%]      [%/min]                      [deg]       [%]      [%/min]

      600.4    100.159      0.00                        675.9    99.909       0.14
      605.6    100.159      0.00                        680.6    99.863       0.10
      610.0    100.161      0.00                        685.2    99.829       0.07
      615.2    100.158      0.00                        691.1    99.800       0.04
      620.1    100.165      0.00                        695.8    99.788       0.03
      625.5    100.158      0.01                        700.6    99.780       0.02
      630.1    100.158      0.00                        705.3    99.775       0.01
      636.1    100.156      0.00                        710.0    99.772       0.01
      641.1    100.158      0.00                        715.9    99.772       0.00
      645.0    100.156      0.01                        720.7    99.774       0.00
      650.1    100.150      0.02                        725.4    99.772       0.00
      655.3    100.137      0.06                        730.1    99.775       0.00
      660.5    100.106      0.11                        736.0    99.777       0.00
      665.8    100.049      0.17                        740.8    99.777       0.00
      671.0     99.975      0.18                        745.4    99.778       0.00
                                                        750.0    99.777       0.00
```

Sample: RUN#54
Size: 77.72 mg
Rate: 05C/MIN
Program: TGA Analysis V2.0

TGA

Date: 29-Aug-85 Time: 15:58:29
File: PEL-COKE.51 TLYNCH.06
Operator: JT
Plotted: 30-Aug-85 8:11:35



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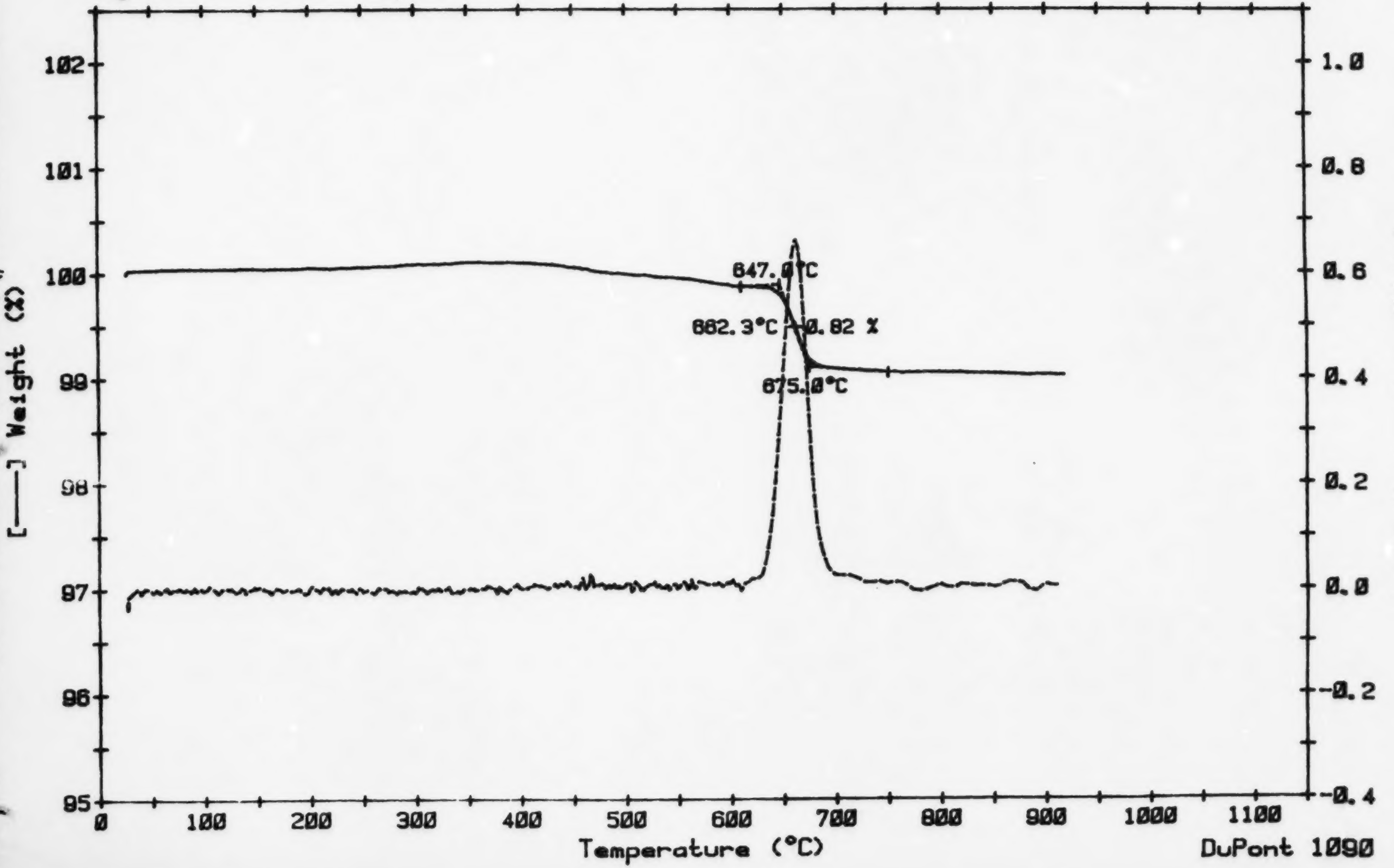
```
Sample: RUN#54                                       Date: 29-Aug-85    Time: 15:58:29
Size: 77.72 mg                                       File: PEL-COKE.51 TLYNCH.06
Rate: 05C/MIN                                        Operator: JT
Program: General Analysis V1.0                       Plotted:  4-Sep-85    11:13:44

    Temp       Sig-A      Deriv-A                         Temp       Sig-A      Deriv-A
    [deg]      [%]        [%/min]                         [deg]      [%]        [%/min]

    400.1     100.077       0.00                          500.1     100.031       0.00
    405.0     100.076       0.00                          505.0     100.027       0.00
    410.0     100.076       0.00                          510.2     100.021       0.00
    415.1     100.076       0.00                          515.0     100.021       0.00
    420.0     100.077       0.00                          520.0     100.015       0.00
    425.2     100.075       0.00                          525.1     100.013       0.01
    430.2     100.072       0.00                          530.1     100.008       0.00
    435.1     100.071       0.00                          535.0     100.005       0.00
    440.3     100.068       0.00                          540.1     100.006       0.00
    445.2     100.066       0.00                          545.1     100.004       0.00
    450.1     100.063       0.00                          550.2     100.000       0.00
    455.0     100.060       0.00                          555.1      99.996       0.00
    460.1     100.058       0.00                          560.0      99.991       0.00
    465.1     100.057       0.00                          565.2      99.988       0.00
    470.2     100.054       0.00                          570.2      99.985       0.00
    475.1     100.049       0.00                          575.0      99.981       0.00
    480.0     100.040       0.00                          580.2      99.973       0.00
    485.0     100.037       0.00                          585.1      99.972       0.00
    490.2     100.032       0.00                          590.0      99.969       0.00
    495.0     100.032       0.00                          595.1      99.963       0.00
                                                          600.1      99.958       0.00
```

```
Sample: RUN#54                                        Date: 29-Aug-85   Time: 15:58:29
Size: 77.72 mg                                        File: PEL-COKE.51 TLYNCH.06
Rate: 05C/MIN                                         Operator: JT
Program: General Analysis V1.0                        Plotted:  4-Sep-85   11:36:38

    Temp       Sig-A      Deriv-A                  Temp       Sig-A      Deriv-A
   [deg]        [%]       [%/min]                 [deg]        [%]       [%/min]

    600.1      99.959       0.00                   675.3      99.381       0.12
    605.6      99.956       0.00                   681.1      99.332       0.07
    610.3      99.952       0.00                   685.7      99.312       0.04
    615.9      99.951       0.00                   690.3      99.289       0.03
    620.8      99.951       0.00                   695.0      99.287       0.01
    626.0      99.951       0.01                   700.9      99.285       0.01
    630.1      99.946       0.02                   705.6      99.281       0.01
    635.5      99.932       0.05                   710.3      99.279       0.00
    641.0      99.906       0.09                   716.1      99.278       0.00
    645.1      99.871       0.14                   720.8      99.276       0.00
    650.7      99.803       0.21                   725.6      99.277       0.00
    656.1      99.705       0.26                   730.2      99.273       0.00
    660.0      99.627       0.26                   736.1      99.273       0.01
    666.1      99.503       0.21                   740.8      99.269       0.00
    670.7      99.431       0.15                   745.4      99.269       0.00
                                                   750.1      99.270       0.00
```

Sample: RUN#47
Size: 51.60 mg
Rate: 05C/MIN
Program: General Analysis V1.0

TGA

Date: 4-Sep-85 Time: 12:06:39
File: PEL-COKE.64 TLYNCH.06
Operator: JT
Plotted: 4-Sep-85 14:13:44



```
Sample: RUN#47                                       Date:  4-Sep-85   Time: 12:06:39
Size: 51.60 mg                                       File: PEL-COKE.64 TLYNCH.06
Rate: 05C/MIN                                        Operator: JT
Program: General Analysis V1.0                       Plotted:  4-Sep-85   14:23:49

     Temp       Sig-A      Deriv-A                        Temp       Sig-A      Deriv-A
     [deg]       [%]      [%/min]                         [deg]       [%]      [%/min]

     400.0     100.097       0.00                         500.0      99.988       0.01
     405.2     100.095       0.00                         505.1      99.977       0.00
     410.0     100.097       0.00                         510.2      99.977       0.00
     415.1     100.087       0.00                         515.1      99.973      -0.01
     420.2     100.083       0.00                         520.1      99.977       0.00
     425.1     100.079       0.00                         525.2      99.969       0.00
     430.0     100.078       0.00                         530.2      99.961       0.00
     435.1     100.074       0.00                         535.0      99.959       0.00
     440.0     100.064       0.00                         540.0      99.953       0.00
     445.1     100.058       0.01                         545.2      99.955       0.00
     450.1     100.048       0.00                         550.2      99.950       0.00
     455.2     100.048       0.00                         555.1      99.942       0.00
     460.2     100.035       0.01                         560.0      99.940      -0.01
     465.0     100.035       0.01                         565.1      99.932       0.00
     470.1     100.017       0.01                         570.0      99.926       0.01
     475.1     100.012       0.00                         575.1      99.913       0.00
     480.0     100.002       0.00                         580.1      99.907       0.00
     485.2      99.998       0.00                         585.2      99.899       0.00
     490.0      99.992       0.00                         590.1      99.891       0.00
     495.1      99.990       0.00                         595.0      99.888       0.00
                                                          600.1      99.882       0.00
```

```
Sample: RUN#47                                    Date:  4-Sep-85   Time: 12:06:39
Size: 51.60 mg                                    File: PEL-COKE.64 TLYNCH.06
Rate: 05C/MIN                                     Operator: JT
Program: General Analysis V1.0                    Plotted:  4-Sep-85   14:42:03

      Temp      Sig-A      Deriv-A                      Temp      Sig-A      Deriv-A
      [deg]      [%]       [%/min]                      [deg]      [%]       [%/min]

      600.1     99.882       0.00                       675.4     99.174       0.14
      605.2     99.874       0.00                       681.1     99.130       0.07
      610.2     99.866      -0.01                       685.6     99.109       0.04
      615.7     99.866       0.01                       690.2     99.098       0.02
      621.1     99.864       0.01                       696.1     99.087       0.01
      625.9     99.860       0.01                       700.8     99.085       0.01
      631.0     99.859       0.01                       705.5     99.081       0.01
      635.0     99.855       0.02                       710.2     99.076       0.01
      640.5     99.841       0.06                       716.0     99.070       0.01
      646.0     99.804       0.13                       720.7     99.066       0.01
      650.1     99.758       0.20                       725.4     99.062       0.01
      655.6     99.657       0.30                       730.0     99.060       0.00
      661.0     99.519       0.36                       735.9     99.060       0.01
      666.1     99.376       0.34                       740.5     99.058       0.01
      670.9     99.254       0.25                       745.1     99.054       0.00
                                                        750.9     99.050       0.00
```